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DEVELOPMENT OF A NICKEL BASE ALLOY SHEET FOR HIGH TEMPERATURE APPLICATION

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Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
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FOREWORD

21) Reporton

This report was prepared by Chance Vought Corp., Aeronautics and Missiles Division, under USAF Contract No. AF 33(16)-7999. This contract was initiated under Project No. 7351, Metallic Materials", Task No. 735105, "High Strength Metallic Materials". The work was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Captain L. F. Bubba, succeeded by Lt. F. L. Krempski, acting as project engineer.

This report covers work conducted from 15 March 1961 to 15 July 1962.

Mr. H. Greenewald, Jr., was the Chance Vought principal investigator. Mr. T. J. Riley cooperated in the research and in the preparation of this report. Mr. R. Calvert was in charge of all mechanical tests.

In addition to the authors, Mr. John Freche and Mr. William Waters of the Lewis Research Center, MASA, Cleveland, Ohio, have made substantial contributions to the work on the NASA TaZ8 alloy.

FOREWORD

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In addition to the authors, Mr. John Freche and Mr. William Waters of the Lewis Research Center, NASA, Cleveland, Ohio, have made substantial contributions to the work on the NASA TaZ8 alloy.

ABSTRACT

The objective of this contract was to develop 15 to 30 mil nickel alloy sheet having 50,000 psi tensile strength at 1900°F, having good corrosion (oxidation) resistance, and good ductility. This objective was essentially attained by developing a new process of directly rolling thin cast slabs of nickel base alloy into sheet on a specially designed rigid rolling mill.

Two pre-existing nickel base casting alloys and a series of experimental compositions obtained by modifying the two starting alloys were initially investigated in the cast condition in this program. The two starting alloy compositions were Inco 713c developed by International Nickel Co. and the TaZ8 alloy developed by Lewis Research Center of NASA. Of the new experimental compositions, TaZ8 alloy and No. 429 alloy have 1900°F tensile strengths exceeding 50,000 psi at 1900°F in the as cast condition. Inco 713c has a tensile strength of about 40,000 psi in the as cast condition.

Procedures were developed on the rigid mill for hot rolling both Inco 713c and TaZ8 alloy into 15 to 30 mil sheet. Heat treatment procedures were developed for both alloys which enabled them to meet contract target strength properties. The maximum room temperature ductility obtained in rolled and heat treated Inco 713c sheet was 20% elongation compared to a maximum of 5% elongation for the rolled TaZ8 alloy. The good room temperature ductility of hot rolled Inco 713c was further indicated by the fact that this hot rolled sheet was cold rolled from 20 mil down to 3 1/2 mil foil. The oxidation resistance of TaZ8 alloy is adequate for limited periods of time at 1900 F; that of No. 429 alloy is substantially better; and that of Inco 713c is best of the three.

This technical documentary report has been reviewed and is approved.

I. Perlmutter

& Perlmitte

Chief, Physical Metallurgy Branch Metals and Ceramics Laboratory Directorate of Materials and Processes

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I. INTRODUCTION

There is a critical need for oxidation resistant metal structures having usefully high tensile strength at temperatures approximating 2000°F. Existing nickel base superalloy sheet materials have adequate oxidation resistance at these temperatures but have relatively low tensile strength properties. Refractory metals have adequate strength at these temperatures but are subject to rapid destruction by oxidation attack. This program has been devoted to solving this problem by developing nickel base sheet materials having improved strength in the 1900-2000°F temperature range.

The primary objective of this program was to develop 15 to 30 mil thick nickel base alloy sheet with short term tensile strength of 50,000 psi at 1900°F, adequate corrosion (oxidation) resistance, and ductility. These target properties represent a substantial improvement over the best commercially available nickel alloy sheet, René 41. René 41 sheet has a tensile strength of 50,000 psi at 1700°F and only about 20,000 psi at 1900°F.

The secondary objective of this program was to demonstrate the feasibility of producing improved nickel alloy sheet in sizes up to $10" \times 48"$. Another purpose of this program was to determine the feasibility of directly rolling sheet from relatively thin cast slabs of high strength superalloys.

The original objectives of this program have been essentially met by two different alloy sheet materials. These two materials are: (1) Inco 713c and (2) NASA TaZ8 alloy sheet. In rolled and heat treated sheet form the Inco 713c sheet has better ductility and better oxidation resistance at 1900°F than the NASA TaZ8 alloy sheet. Both materials have about the same 1900°F tensile strength. Therefore, on balance, the Inco 713c rolled and heat treated sheet must be considered the more desirable engineering material insofar as short time tensile properties are concerned.

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II. DESCRIPTION OF THE PROGRAM

The program objective was pursued in three ways as follows:

- (a) Improvement by changes in chemical composition.
- (b) Improvement through mechanical working by rolling of the sheet.
- (c) Improvement by heat treatment of the rolled sheet.

The alloys used in this program were formulated by melting in a plasma-resistance furnace. The molten alloy was then cast into hot ceramic molds to directly form cast sheet blanks about 1/8 inch thick. All alloys cast were evaluated in the as cast condition. Alloys having useful properties in the as cast condition were rolled into sheet. This rolled sheet was then evaluated in both the as-rolled and heat-treated conditions.

III. EXPERIMENTAL PROCEDURES AND TESTS

A. Processing Techniques

1. Melting and Casting Techniques

All metal evaluated in this program was melted and cast in the plasma-resistance furnace developed previously at Vought. All castings used in this program were cast in hot ceramic molds. This molding and casting technique permits the casting of relatively large, thin sheets of nickel base superalloys, as well as a wide range of other alloys. Most of the cast sheets used for the program were about 0.1 inch thick and approximately 5" x 8" in size. Larger sized cast sheets approximately 8" x 16" were also made. This larger sheet represents the largest mold which can be made on the existing molding press at Vought, but is not believed to represent any maximum size limitation of the process as a whole. It was subsequently shown that sheets approximately 10" x 16" x .025" can be rolled from the 5" x 8" cast sheet. Efforts have been made to roll thicker cast slabs and slices from ingots in this program. These efforts have been unsuccessful, demonstrating the practical need for casting the starting sheet in thicknesses approximately 0.1 inch. A more detailed description of the melting and casting techniques used is given in Appendix A, Melting and Casting Procedures and Equipment.

2. Rolling Procedures

a. Sub-Contract Rolling - Metals and Controls

Virtually all the rolling work done at Metals and Controls Division, Texas Instruments, Inc., Attleboro, Massachusetts, was done on two rolling mills: (1) a two high mill with 7 inch diameter rolls; and (2) a two high mill with 20 inch diameter rolls. Most of the rolling done was performed at room temperature with periodic intermediate annealing heat treatments at temperatures of from 2000°F to 2150°F.

One test was made rolling the as cast NASA alloy at 1500°F and another test was made rolling the NASA alloy at 1500°F after the sheet had been previously cold rolled and annealed at 2150°F. All cold rolling was performed with reductions in thickness per pass of only one to two thousandths of an inch. All hot rolling was done with reductions per pass of under 0.005 inch, although this was much harder to control. Hardness readings were usually taken on the sheet after

each pass through the mill and the sheet was measured for thickness with a micrometer after each pass. The design and construction of the rolling mills is such that it was not possible to determine the actual applied rolling loads. Most of the specimens rolled were about 2 inches wide by 4 inches long. In addition to rolling at room temperature and at 1500°F. the NASA alloy was rolled at liquid nitrogen temperature (-320°F). Rolling tests were performed on the Ta-C modified Inco 713c (Melt No. 388), the Inco 713c and the NASA alloy compositions to determine the work hardening characteristics of the alloys during cold rolling. The rolled alloys were also subjected to a variety of heat treatments to determine their annealing characteristics and metallographic specimens were taken as appeared advisable.

b. In House Rolling - Chance Vought

Difficulties encountered in rolling these alloys at Metals and Controls resulted in Vought designing and constructing a unique rigid rolling mill in support of this program. Future reference to the "rigid" rolling mill will connotate the Vought constructed mill. A detailed description of this rolling mill and the major factors affecting its design are given in Appendix B, The Rigid Rolling Mill. The following procedure was used in rolling sheet on the rigid mill in this program.

- 1. With the rolling mill in the two high configuration, set the gap between the two rolls so that the cast sheet to be rolled would just fit.
- Preheat the cast sheet to from 1750°F to 2150°F and immediately put the hot sheet through the rolling mill.
- 3. Replace the rolled sheet in the preheat furnace and adjust the roll gap downward by a predetermined amount of from 3 to 6 mils. Remove the sheet from the furnace, pass it through the rolls, and replace the sheet in the furnace to reheat.

 Note the separating force experienced by the mill by reading the strain gauge recorder.
- 4. Repeat this cycle until the sheet is reduced to from 40 to 60 mils.

- 5. Convert the rolling mill to the 4 high configura-
- 6. Resume hot rolling, still using 3 mils reduction per pass as per the procedure in 3 above. Continue this until a sheet thickness of about 15 mils is reached.

3. Heat Treatment Procedures

The first heat treatment attempted on cast NASA TaZ8 alloy was done in air at 2200°F. This resulted in very serious oxidation and scaling of the specimens. All subsequent heat treatment was done under an inert atmosphere or by encapsulating the specimens in stainless steel or Inconel. The encapsulation procedure worked very well provided there were no leaks in the stainless steel or Inconel box and provided the stainless steel box did not scale through. Both leaks in the welds and scaling through of boxes and of welds have given trouble at times with the encapsulation process. Also, it is difficult to control cooling rates in the specimen while it is encapsulated. Therefore, the normal heat treatment procedure is to heat the specimen in an Inconel retort under a low flow rate of argon and with a slight positive pressure of argon maintained at all times in the retort. This procedure has worked very well for all alloys and for all heat treatments used in this program.

B. Evaluation Techniques

1. Room Temperature Tests

The test specimen used for room temperature tests is shown in Fig. 1. Specimens were loaded in a universal test machine with a loading accuracy of 1% of the applied load.

Strain was measured over a one inch gage length with a Baldwin microformer type extensometer complying with ASTM method E 83-57T strain accuracy classification B-2. The load and the strain were plotted autographically on a Baldwin Load-Strain recorder. The specimens were strained at a rate of 0.005 ± .002 inches per inch per minute through yield. After yield a strain rate of .05 inches per minute head travel was used. Total elongation by caliper was determined over a one inch gage length.

2. Elevated Temperature Tests

The test specimen configuration is shown in Fig. 2. Specimens were loaded in a universal test machine with a

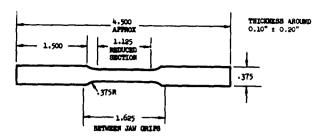
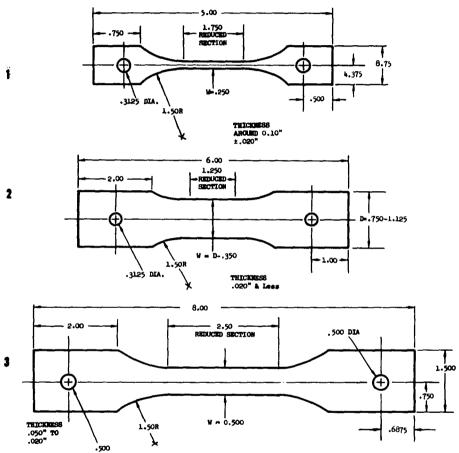


Figure 1. Sheet Tensile Specimens used for Room-Temperature Tests.



- 1. Tensile Specimen used for All Elevated Temperature Tests on As-Cast Sheet Prior to Melt 428.
- 2. Tensile Specimen used for Most Elevated Temperature Tests on Rolled Sheet, Performed using Resistance and Radiant Heat.
- Tensile Specimen used for Elevated Temperature Tests, Performed in a Tube Furnace.
- * All Dimensions are Approximate.

Figure 2. Typical Elevated Temperature Tensile Specimens.

loading accuracy of 1% of the applied load. Specimen heating was accomplished with either quartz lamps or resistance heating.

The power source for both methods of heating was an ignitron power controller. For resistance heating the output of the power controller was supplied to a Kirkhof welding transformer which in turn supplied power directly to the test specimen.

The method of attaching the power leads to the test specimen for resistance heating is shown in Fig. 3. For quartz lamp heating, controlled voltage was supplied to two lamp reflector assemblies, each containing five lamps, placed equal distances from each surface of the specimen, Fig. 4.

Specimen temperatures were sensed with 36 gauge chromelalumel thermocouples spot welded to the test specimen and recorded on a Brown strip chart multipoint recorder.

Specimens were loaded at a rate of 16,000 psi per minute. Total elongation was determined by caliper over a one inch gage length.

A more detailed description and discussion of the elevated temperature testing procedure is given in Appendix C, Elevated Temperature Tensile Procedure.

3. Oxidation Testing

a. Determination of Weight Change after Exposure to Still Air at High Temperature

The specimen of sheet to be oxidation tested is belt sanded down to clean metal on all sides. The specimen is accurately measured to determine total surface area and weighed. The specimen is placed in a sillimanite crucible of known weight and the crucible with the specimen in it is placed in a globar furnace with a still air atmosphere for a predetermined length of time. After exposure to the air in the furnace, the crucible with the specimen in it is removed from the furnace and allowed to cool in air with a lid over the crucible. It is necessary to have a lid over the specimen while it is cooling since the oxide scale formed on some alloys spalls off vigorously upon cooling and is lost. After the specimen has cooled, the net weight of the specimen plus the oxide scale is determined and the weight gain in terms of milligrams per square centimeter of area is

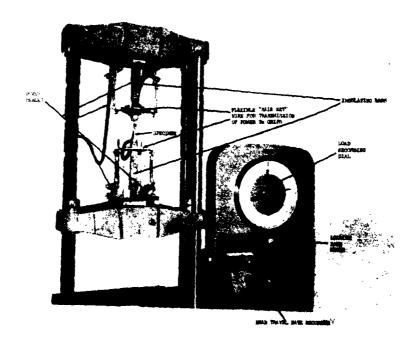


Figure 3. Typical Tensile Test Setup for Resistance Heating.



Figure 4. Relationship of Specimen to Quartz Lamps Used for Radiant Heat Tensile Tests.

calculated. The oxide scale on the specimen is removed by hard rubbing with a towel. The specimen is reweighed and the weight loss in terms of milligrams per square centimeter is determined.

These tests give an indication of the relative oxidation resistance of the metals and alloys tested. They do not take into account important factors such as:

- 1. Depletion of surface layers of the alloy with consequent weakening of the structure.
- 2. Intergranular attack.
- Diffusion of oxygen and nitrogen into the alloy with consequent damage to properties and effect on weight gain measurement.
- 4. Volatility of some of the oxides which might be formed on these alloys, affecting the weight gain measurements.
- 5. Effects of air velocity during oxidation.
- 6. Effects of pressures other than atmospheric.
- 7. Effects of temperature cycling.

The results of the oxidation tests performed in this program should therefore be taken as an indication of relative values for certain particular conditions.

More detailed testing should be related to a particular end use environment.

b. <u>Identification of Oxidation Products by X-ray</u> Diffraction

A General Electric XRD-5 X-ray diffractometer, equipped with a cobalt tube was used to determine the identity of the oxides formed on these alloys. The SPG-2 detector tube was used with the scaler-recorder, and pulse height discrimination was not used. A Fe₂O₃ filter, double layer, was used to eliminate the K-beta radiation.

The oxides were ground up to less than 325 mesh using an agate pestle and mortar. This powder was then pressed into the powder specimen holder and mounted for diffraction patterns. The diffraction pattern covered the region of 60 to 1470 theta. The interpretations of the patterns were based on the ASTM File of X-ray Powder Data.

4. Metallographic Examination

a. Optical Metallography

Conventional mounting and polishing techniques were used to prepare metallographic specimens for examination in this program. The following etching solutions were used to etch the specimens for examination on the optical and electron microscopes:

- (1) Conc. HCl & FeCl3 (etchant #1)
- (2) A mixture of Conc. HCl & H₂SO₄ (etchant #2)
- (3) Conc. HCl & drops of H_2O_2 (30%), (etchant #3)
- (4) NASA electrolytic etch (etchant #4)
 - 4 parts H₂0
 - 4 parts Glycerine
 - 2 parts Conc. HNO3
 - 1 part Conc. HF

b. Electron Microscope Metallography

The electron microscope replicas used in this report were prepared in accordance with the following technique:

Cellulose acetate was softened with acetone and placed on the polished and etched surface to be replicated, allowed to dry to stiffness, and then carefully removed.

The negative plastic replica produced in this step was placed in a vacuum evaporator and shadowed with chromium at an angle of 30°, the carbon was deposited normal to the replica surface until the desired thickness was obtained.

The double replica was then removed from the evaporator, cut into small squares. The small sections of the double replica were then placed in a dish of acetine until the plastic dissolved, leaving a chromium shadowed carbon positive replica of the original surface. This positive replica was studied on a Norelco Model 100B electron microscope.

IV. EXPERIMENTAL DATA AND DISCUSSION

A. Alloy Improvement by Varying Chemical Composition

1. Choice of Starting Alloy Composition

The objective of this contract was to obtain nickel base alloy sheet of improved elevated temperature tensile properties. At the time of the initiation of this contract in 1961, the nickel base alloys having the best elevated temperature tensile strength were all casting alloys and were not available in sheet form. Two of the best of these casting alloys were selected as starting compositions for this investigation. One alloy selected was Inco 713c. This alloy is among the best of the commercially available superalloys, and has been proven as a casting alloy in many applications over a period of several years. The other alloy selected was the NASA TaZ8 alloy. This alloy had the highest tensile strength at 1900°F of any superalloy on which data was available. This alloy was a new and experimental alloy having great promise, but was not commercially available and had no previous history as an engineering material. The standard compositions and previously reported room temperature and 1900°F tensile strengths of these two starting alloys are given in Table 1.

These two starting alloys were modified in this program by making changes in chemical composition, by rolling, and by heat treatment. All three of these techniques resulted in substantial changes in the metallurgical structure of both alloys. For complete casting data refer to Appendix D.

2. Modification of Starting Alloy Compositions to Improve As-Cast Properties

a. Modification of Inco 713c

The primary strengthening mechanism in Inco 713c is the precipitation of gamma prime. This is a complex intermetallic composed of nickel, aluminum, and titanium. The precipitation of titanium and other carbides is also believed to contribute to the good high temperature strength of Inco 713c. Figure 5 shows the structure of Inco 713c gamma prime precipitates in cast thin sheet. Figure 6 shows both the gamma prime precipitate and the titanium carbide

precipitate occurring in the grain boundary of Inco 713c cast into a relatively thick section. The titanium carbide precipitate has a marked tendency to occur along the grain boundaries in the as cast Inco 713c, and very little carbide precipitate is noted elsewhere in the structure.

In this program efforts were made to modify substantially the structure of the as cast alloy by increasing the amount of gamma prime producing elements and by increasing the amount of carbide producing elements in the alloy. In the first case, aluminum and titanium contents were increased either together or separately. In the second case, carbon content was raised and strong carbide forming elements such as tantalum and tungsten were added simultaneously. Table 2 summarized the various modified Inco 713c compositions investigated. All of these alloys had low ductility at room temperature and all were difficult to machine.

The two melts containing very high percentages of aluminum were extremely brittle, and both proved to be unmachinable because of this brittleness. Inco 713c modified with smaller aluminum additions plus a small titanium addition was far less brittle than the high aluminum alloys, but even this alloy had such poor ductility that several tensile specimens were lost in the machining process. It should be pointed out here that all of the modified Inco 713c alloys were substantially more difficult to machine than Inco 713c, either because of a tendency to break during machining because of low ductility or because of the hardness of the precipitated carbides in the alloys. Later in the program, machining techniques were developed for making tensile bars which could have saved a number of the tensile bars lost during machining at this stage in the program. The loss of a number of specimens during machining accounts largely for the limited amount of tensile data available for these alloys.

Three efforts were made to strengthen Inco 713c by increasing the nickel aluminide precipitate. These efforts are contained in Melt Nos. 430, 431, and 438. Melt Nos. 430 and 431 were so brittle that no usable tensile bars were obtained. Melt 431 was so brittle that the casting broke up into small pieces during vapor blasting. Melt No. 438 was much less brittle; however, only one usable tensile bar was obtained from Melt No. 438 and the rest broke during machining.

Unfortunately, the sole surviving tensile bar was a room temperature bar and was not an elevated temperature test bar. Limitation of available time prevented the making of another melt of this composition, so 1900°F tensile strength was not determined on any of the aluminum and titanium modified Inco 713c alloys. Figures 7, 8 and 9 show the microstructures of these three alloys in the as cast condition. All three structures show a relatively coarse structure which might be improved by heat treatment. However, the observed properties did not appear to warrant this additional investigation.

A total of eleven different usable melts were made of carbide modified Inco 713c. One melt (Melt No. 395) was attempted having a very high tantalum-carbon content, and this melt reacted with the crucible to such an extent that no usable casting resulted. Another melt, No. 403 resulted in cast-sheet having excessive gas porosity which made it unusable for tensile bars. Of the eleven usable melts, Melt No. 434 was so brittle that all tensile test bars broke during machining. All of the remaining melts, with the exception of No. 429, had about the same room temperature ductility as the as cast NASA TaZ8 alloy. The 1900°F tensile strengths of all compositions tested were equal to or superior to that of Rene 41, but only No. 429 had 1900°F strength meeting program target requirements. The microstructures of twelve alloys cast are given in Figures 10 through 21. All of these microstructures show substantial amounts of precipitated carbides and most show microstructures substantially different from either Inco 713c or NASA TaZ8 alloy.

There is much more variation in 1900°F tensile strength among these alloys than can be explained by the variation in the amounts of carbide precipitate observed in the microstructures. For example, Melt No. 429 has about 50% greater 1900°F tensile strength than Melt No. 435 although the total amount of carbide precipitate in the two alloys appears to be about the same. Furthermore, the only compositional difference between the two alloys is that 429 has more tantalum and more carbon. It is, therefore, evident that it is not only the quantity of carbide precipitated, but also the composition of the carbide that is important. Furthermore, it is interesting to note that alloys containing up to 1.66% carbon still have room temperature and 1900°F ductility about the same as Inco 713c, which

TARE 1 Standard Compositions

and Strength of INCO 713C & NASA Alloy (AS CAST)

1900*F UNS (pe1) 54,300 37,000 Tensile Properties 5.2 H ROOM TEMPERATURE UTS UTS (pe1) (pe1) 134,400

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NASA(3)

INCO 713C(1,2) Ball.

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Composition

3.5

5.5 6.5

0 1 1 1 9.0 ៥

3.0

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Modified INCO 713c Alloy Compositions and Strength Properties

Q)

TABLE

Melt Composition (Determined by Chemical Analysis)

.o15 9 1.0 S I .25-1.25

102,000

11,000

S

67

8.0

45,400

2.0 2.0 9.0

110,700

119,300 63,600 117,900 108,950 71,600

1.68 1.72 0.78

1.30 1.34 1.55 1.15 0.93 1.35

0.89 0.83

₹.0

0.07

0.19

3.6 3.6 3.47 17.27 3.50 3.51 3.11 3.75

10.98 10.83

10.87

63.94

0.29 1.16 1.66 5.46

0.16

0.20

5.59

11.06

64.08

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Zr

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Ta

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ALLOY

MELT No.

14

SEL

ROOM TEMPERATURE 1500°P UTS UYS SEL UTS

10.0

112,700

0.35 9.0 0.52 કે.

0.72 0.17

0.19 0.15 91.0 0.15 91.0 0.25 0.15

40,700 37,000 28,400

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1.5

53,200

1.07 1.54 1.57

0,0

9

24,300

1

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1.54 1.40 2.42 3.12

0.0

37,000 39,400

0.1 1.0 1.0 0.1

82,900 90,500 116,400

1.07

.. 8

1.14

0.16 91.0 0.15 0.19

15.83

4.55

0.17

14.85 4.65 3.89 4.21 3.95

5.07

0.13 0.16

713-C Mod

INCO INCO INCO

434 435 436 437

Jr-W-C Mod

LNCO

1.34 1.22 1.45

7.19

1.15 5.66 0.30

0.13

34,400

;

107,400

1.76

2.68

0.16

1.35

0.23

7.76

5.32 9.65

12.33

0.83 0.15

INCO 713-C Cb-Ta-W-C Mod INCO-713C A1-T1 Mod INCO 713-C

12.20

1

5.83

14.07

0.55

27,100 10.0

2.0

91,600

1.83 1.50

1.41 1.37 1.31

1.26 11.9 5.46

> 1.29 0.40

3.36

6.64

11.09 11.12 11.25 17.30

0.45

84.49 60.84 60.02 50.50 63.59 94.09 66.52

0.17

1.27

0.81

0.13 0.54 1.11 2.45

0.12 7.21

0.31

9.0 0.10

17.75

4.16

7.58

43.87 57.59

10d INCO-713C

Al-T Mod

19.01 9.81 27.64

11.37

0.75 0.18

52.30

INCO 713-C Al Mod

431

LNCO

4.99

9.57 71.71

52.76

12.01

67.54

Ta-C Mod INCO 713-C Ta-C Mod INCO 713-C 2(Ta-C) Mod INCO 713-C Ta-C-Mo Mod INCO 713-C Ta-C-Cr-W

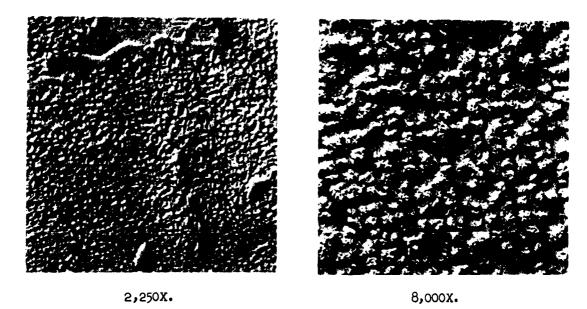


Figure 5. Electron Micrographs of Inco 713c in the As-Cast Condition, Melt 405. Etch No. 1.

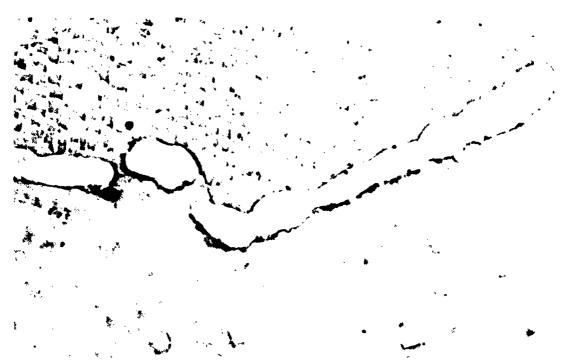
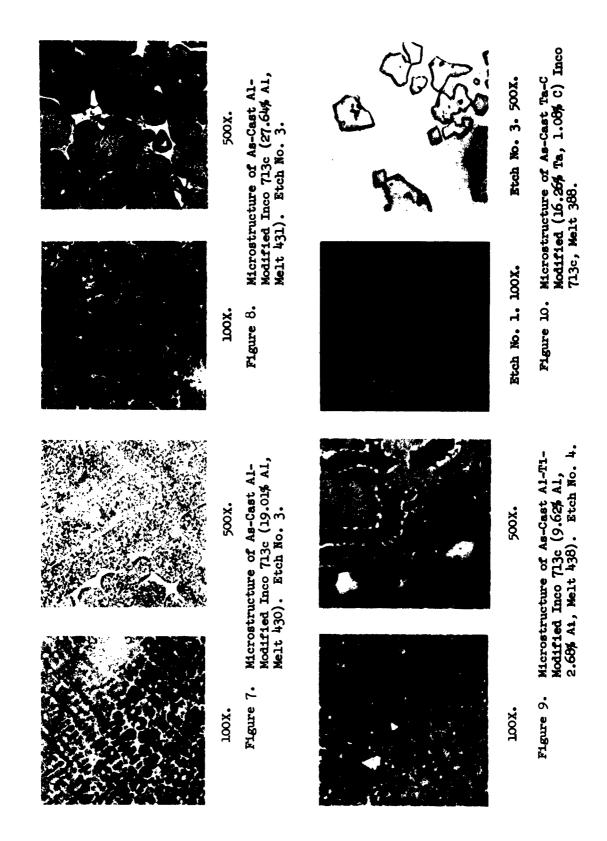
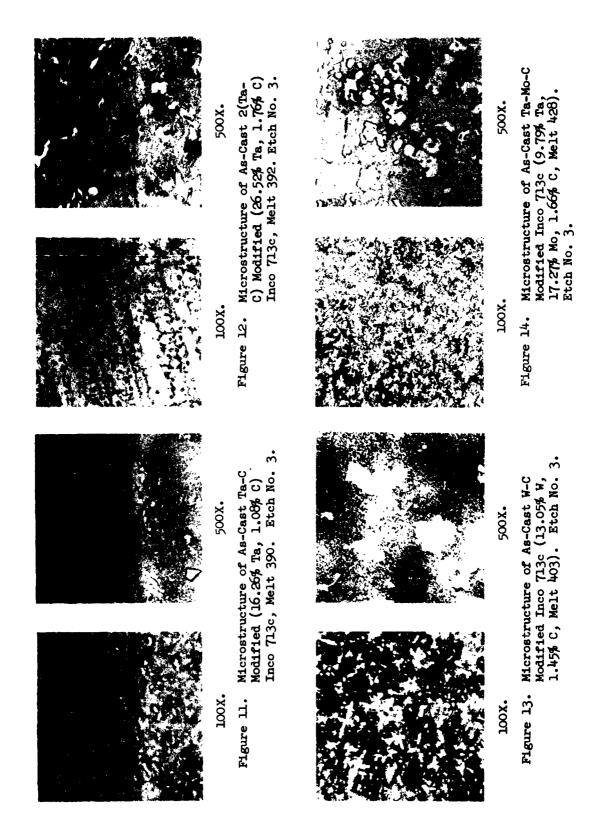
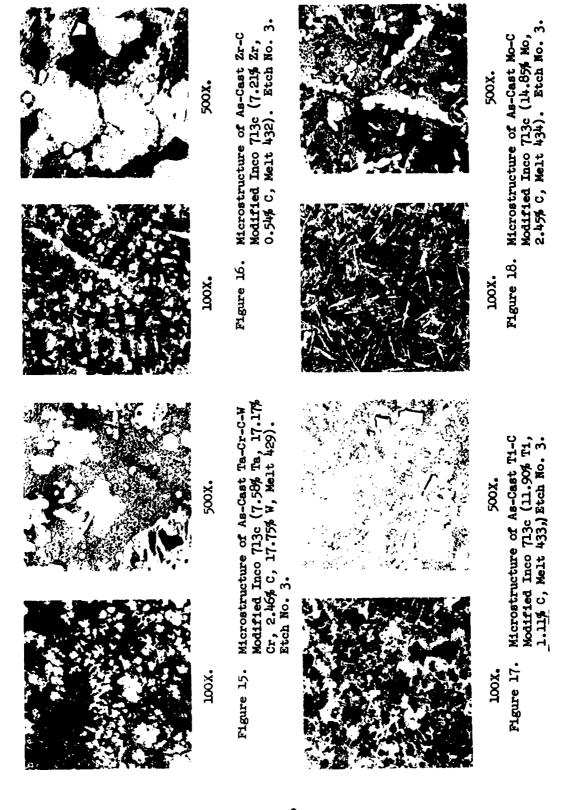
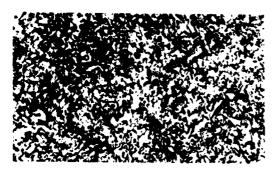


Figure 6. Electron Micrograph Showing TiC Precipitate in a Grain Boundary of As-Cast Inco 713c. 9,000X. Heat No. 56.







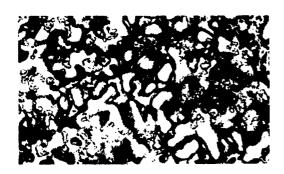


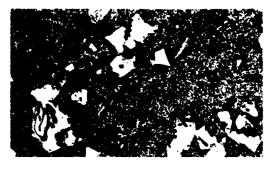


100X.

500X

Figure 19. Microstructure of As-Cast Cr-W-C Modified Inco 713c (17.30% Cr, 15.83% W, 1.14% C, Melt 435). Etch No. 3.

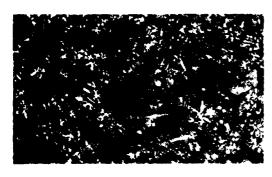


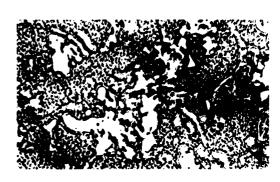


LOOX.

500X.

Figure 20. Microstructure of As-Cast Cr-Ti-C Modified Inco 713c (14.07% Cr, 7.19% Ti, 1.15% C, Melt 436). Etch No. 3.





100X.

500X.

Figure 21. Microstructure of As-Cast Cb-Ta-W-C Modified Inco 713c (3.12% Cb, 0.83% Ta, 7.76% W, 2.66% C, Melt 437). Etch No. 3.

has only about 0.16% carbon. Considering the low atomic weight of carbon and the high molecular weight of the carbides formed, 1.66% carbon corresponds to a high atomic percentage of carbide present. It would therefore appear that an alloy strengthened at high temperature by carbide precipitation is not of necessity brittle.

Accurate determination of the solidus temperature of these complex alloys is very difficult. However, specimens cut from each of these alloys were exposed to a variety of temperatures from 2200°F to 2500°F. The reproducibility of results was poor and so no accurate tabulation is possible. However, it appeared that the alloys showing the lowest strength at 1900°F had the lowest solidus temperature and that Melt No. 429 had a solidus temperature of about 2500°F. This would indicate that further improvement in 1900°F strength will require the development of alloys having higher solidus temperatures. Since equilibrium diagrams are not available for these very complex alloys, further development in this direction must be along rather empirical lines.

Table 3 and Figure 22 show a comparison of the tensile strengths of four alloys at temperatures of 1900°F and above. The data given for the rolled Inco 713c is based on duplicate tensile tests, while the data for the other alloys is all based on single tensile bar results. In spite of the extremely limited number of tests, the available data does appear to fit a logical pattern. Firm conclusions must await the acquisition of further test data.

Table 1 shows that the Inco 713c and NASA TaZ8 alloy compositions are substantially different. Figure 23 shows that as cast Inco 713c and NASA TaZ8 alloys vary substantially in microstructure. Figures 10, 11, and 12 show that Inco 713c with tantalum and carbon additions have as cast microstructures similar to that of the NASA TaZ8 alloy and dissimilar to that of Inco 713c. Figures 24 and 25 show comparisons between the microstructures of cast and heat treated NASA TaZ8 alloy (Melt 385) and the microstructure of cast and heat treated Ta-C modified Inco 713c alloy (Melt 388). Figure 26 shows that relatively large variations in the tantalum and carbon content of Ta-C modified Inco 713c do not greatly alter the microstructure of the alloy. Table 2 shows that the highest 1900°F

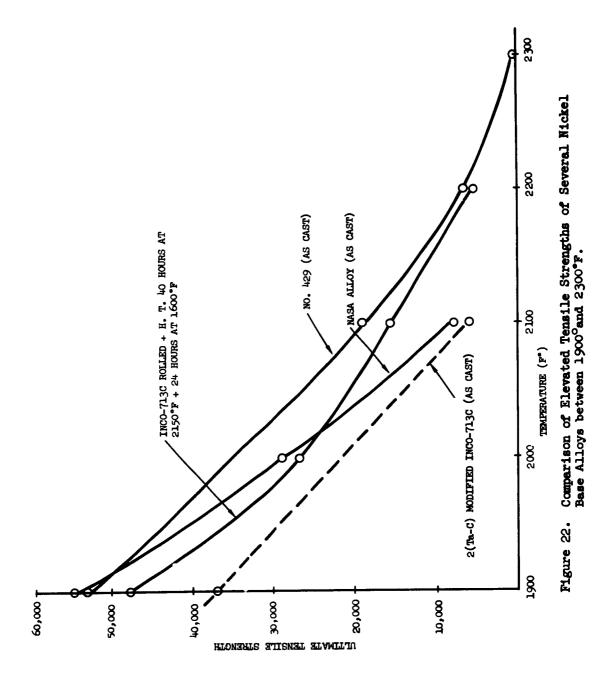
strength in Ta-C modified Inco 713c (Melt 388) is achieved when both tantalum and carbon additions are high. Figure 27 shows a comparison of the microstructure of Inco 713c (Melt 405), Ta-C modified Inco 713c (Melt 395), No. 429 alloy (Melt 457) and NASA alloy (Melt 387) in the as cast condition at 25,000 diameters magnification. Each of the four microstructures is appreciably different from each of the others. The differences between the NASA, Inco 713c, and No. 429 alloys were apparent at lower magnifications. The Ta-C modified Inco 713c (Melt 395) is seen to be different from the NASA TaZ8 alloy at this magnification, whereas optical microscope studies had failed to reveal this difference. The difference between the two alloys appears to be more one of precipitate size than a difference in basic structure. In each instance, the NASA TaZ8 alloy is markedly similar in microstructure to the various Ta-C modified Inco 713c alloys. It appears that the characteristic structure of the NASA TaZ8 alloy is caused by the presence of significant amounts of tantalum and carbon. In both the as cast and heat treated conditions the NASA Taz8 alloy has substantially better 1900°F tensile strength than the Ta-C modified Inco 713c compositions. It is believed that the significant compositional difference here is the presence of 4% tungsten in the NASA Taz8 alloy. Here, as in the No. 429 alloy, the concurrent presence of tantalum, tungsten, and carbon appear to be necessary to the attainment of maximum 1900°F tensile strength. Additional tests beyond the scope of this program will be required to characterize this relationship, or even to prove beyond doubt that it exists.

The No. 429 alloy has a high carbon content. Metal-crucible reactions are a serious problem when this alloy is melted in either a magnesia or a zirconia crucible. Two efforts were therefore made to melt this alloy in a graphite crucible. Both efforts were unsuccessful. In spite of the high carbon content of the No. 429 composition, the alloy dissolved still more from a graphite crucible. This erodes the crucible, changes the composition of the alloy, and raises the melting point of the alloy to a point where it becomes impossible to pour a casting. It would therefore appear advisable to attempt melting this alloy in a water cooled copper crucible, but this was beyond the scope of this program.

TABLE 3

1900°F TO 2300°F COMPARISON OF TENSILE STRENGTHS OF INCO 713C, MOD INCO 713C & NASA ALLOY

ALLOY	MELT	CONDITION	Test Tesperature Op	ULTIMATE TENSILE STRENGTH (ps1)	% Elongation
INCO 713c	502 504 505 505 506 506 506 506 506 506 506 506	As cast As cast Rolled Rolled	1900 1900 2000 2100	15,900 15,000 15,600	4.0 0.0 0.0 0.0 0.0
e (Ta-C) nodified INCO 713c	385	Kolled As cast As cast	2100 2100	37,980 37,980 8,680	11.0 10.0 -
Ta-C-Cr-W modified INCO 713c	429 457 457 457	As cest As cest As cest As cest	1900 2100 2200 2300	53,200 19,000 6,700 418	1.5 7.0 3.0 2.5
KASA	333 334 334	As cast As cast As cast As cast	1900 1900 2000 2100	46,200 53,200 28,900 7,900	31.8



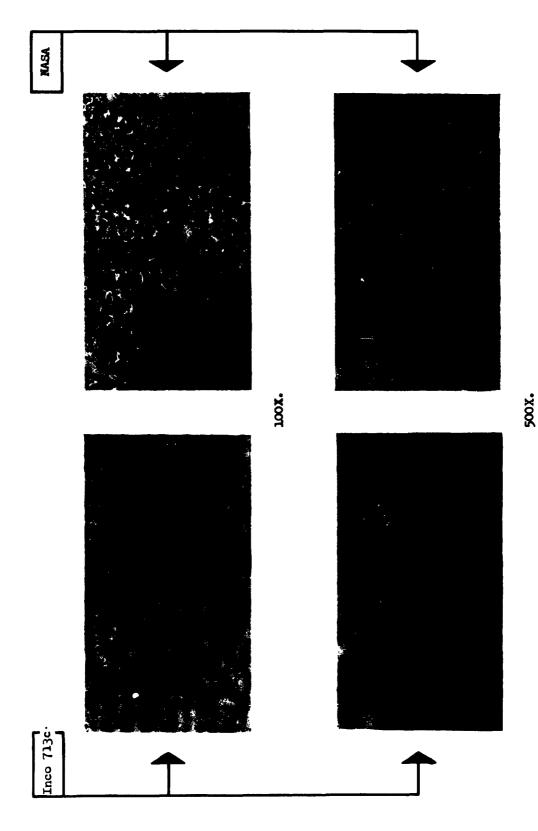
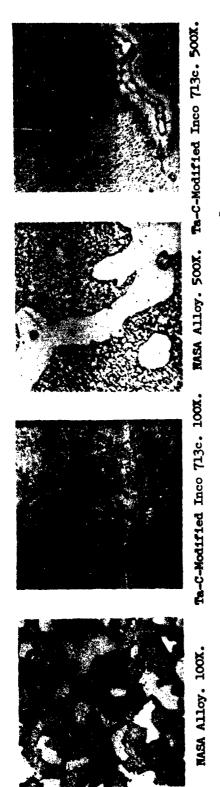
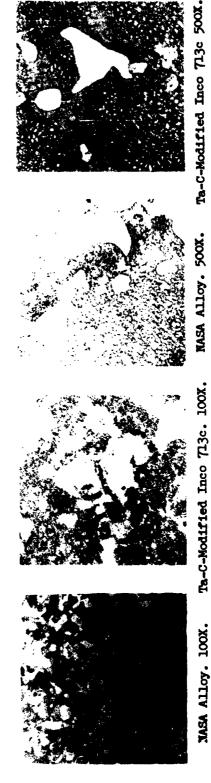


Figure 23. Comparison of Microstructure of As-Cast Inco 713c Sheet with That of As-Cast NASA. Etch No. 3.



16 Hours And Aged At 1500°F For 28 Hours. Etch No. 1. Comparison Of Microstructure Of NASA Alloy, Melt 385, With That Of Ta-C-Modified Inco 713c (16.26% Ta, 1.08% C, Melt 388), Both Heat Treated At 2000°F For Figure 24.



With That Of Ta-C-Modified Inco 713c (16.26% Ta, 108% C, Melt 388), Both Heat Treated At 2200°F For 16 Hours Comparison Of Microstructure Of NASA Alloy, Melt 385 And Aged At 1500°F For 28 Hours. Etch No. 1 Figure 25.

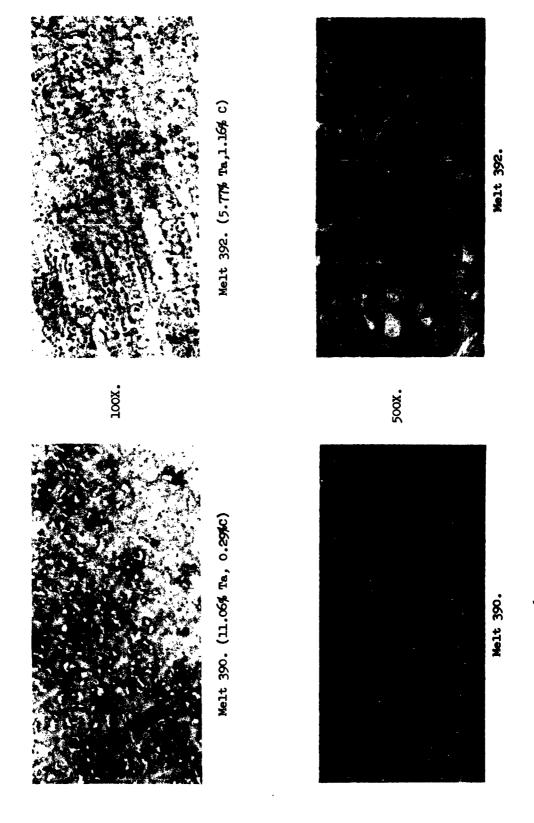


Figure 26. Effects Of Variations In Tantalum And Carbon Additions On The Microstructure Of As-Cast Ta-C-Modified Inco 713c. Etch No. 3.



As-Cast Inco 713C.







Tantalum-Modified Inco 713C, As Cast.





No. 429

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NASA Alloy

Figure 27. Comparison Of As-Cast Microstructure Of Inco 713c (Melt 405), Ta-C-Modified Inco 713c (40.56% Ta, 2.63%C, Melt 395), No. 429 Alloy (Melt 457) And NASA Alloy (Melt 387) As Shown By Electron Micrographs At 25,000X.

b. Modification of NASA Alloy

The as cast 1900°F tensile strength of the NASA TaZ8 alloy met the contract target of 50,000 psi tensile strength at 1900°F from the beginning. However, this alloy has relatively low room temperature ductility and relatively poor oxidation resistance. While the modifications in Inco 713c alloy composition were made in an effort to improve 1900°F tensile strength, the modifications in the NASA alloy composition were made in an effort to improve ductility and oxidation resistance.

Table 4 summarizes the results obtained in all melts made of the modified NASA alloy compositions. Melt Nos. 426 and 427 were made in an effort to improve oxidation resistance of the alloy without diminishing high temperature strength. Both modifications resulted in unacceptably low 1900°F tensile strength so further investigation of these modifications was abandoned. Figures 28 and 29 show the as cast microstructure of these two modified NASA alloys. In both alloys the characteristic NASA alloy structure has been substantially changed.

Melt Nos. 529 through 534 represent NASA alloy modifications designed to improve room temperature ductility. The most significant changes in composition in this series were the addition of small amounts of iron. These modifications seemed to result in a small increase in room temperature ductility, but at the cost of a substantial reduction in 1900 F tensile strength. The effects of these minor changes in composition on microstructure appear to be quite great. Figure 30 shows the microstructures of this series of modified NASA alloys. Some of these structures appear to more closely approach the structure of Inco 713c than that of normal NASA alloy. Since these melts were not chemically analyzed, it is possible that higher than usual melt losses of key elements, such as tantalum, have had a controlling effect on the microstructure and properties rather than the relatively minor additions made deliberately. In any case, none of these melt compositions appear promising as improvements over the Taz8 NASA alloy.

3. Modification of Inco 713c Alloy to Improve Rolling Properties

The NASA alloy, and the first Inco 713c alloy cast, rolled satisfactorily on the rigid rolling mill. After the first

Inco 713c sheet to be rolled had been evaluated and found to be promising, additional melts of Inco 713c were made. These additional Inco 713c castings were found to be substantially more difficult to roll than the original melts of Inco 713c.

A series of melts was made varying slightly the chemical composition of Inco 713c in an effort to obtain cast sheets having the rollability of the first Inco 713c castings made. (Refer to Table 5). The chemical composition was varied by adding trace elements, by increasing the content of the strengthening elements within specification limits, and by adding small percentages of tungsten. Melt Nos. 547, 548 and 549 represented modifications of Inco 713c by the addition of trace amounts of boron, zirconium, and cerium. None of these modified alloys showed any improvement in rolling characteristics. Melt Nos. 550 and 551 were made increasing the tantalum, aluminum and molybdenum contents to near the maximum allowable under the Inco 713c specification. Melt Nos. 552 and 553 were made with small tungsten additions plus increasing the tantalum and molybdenum contents to near maximum Inco 713c specification limits. Melt No. 550 rolled very well and the tensile properties of the rolled and heat treated sheet were approximately the same as those of the first Inco 713c sheet rolled. The castings from Melt Nos. 551 and 553 rolled as well as Melt 550. The castings from Melt No. 552 rolled well but showed a somewhat greater tendency toward edge cracking than Melt 550.

The most serious problem in rolling Inco 713c has been the tendency on the part of some Inco 713c castings to crack at the grain boundaries during the later stages of hot rolling (see Figure 31). Increasing the content of the strengthening elements (Ta, Mo, and Al) apparently strengthens the grain boundaries to the point where they cease to fail during rolling. This also has the effect of increasing 1900°F tensile strength. The reason for adding small percentages of tungsten to the last two melts was that the first good melts of Inco 713c were made in the same crucible immediately after a high tungsten melt and are thought to have contained small percentages of tungsten as a result. Although these melts were not specifically analyzed for tungsten the analysis did report "significant quantities" of tungsten present.

TABLE 4

Modified MASA Alloy Composition & Strength (AS CAST)

Composition

Mechanical Properties

N1 Ta Cr A1 W V Zr C 59.63 7.15 17.68 4.56 3.50 3.40 1.83 0.73 0.18 57.15 7.17 17.15 8.56 3.10 3.18 1.57 0.73 0.25 67.2 7.9 7.0 6.0 5.0 4.0 2.5 0.09
17.00 4.50 3.40 1.83 17.15 8.56 3.10 3.18 1.57 7.0 6.0 5.0 4.0 2.5
cr A1 W Wo V 17.68 4.56 3.50 3.40 1.83 17.15 8.56 3.10 3.18 1.57 7.0 6.0 5.0 4.0 2.5
cr Al W No 17.68 4.56 3.50 3.40 17.15 8.56 3.10 3.18 7.0 6.0 5.0 4.0
cr A1 W 17.68 4.56 3.50 17.15 8.56 3.10 7.0 6.0 5.0
cr A1 17.68 4.56 17.15 8.56 7.0 6.0
cr 17.68 17.15 7.0
N1 Ta Cr 59.83 7.15 17.68 57.15 7.17 17.15 67.2 7.9 7.0
N1 Ta 59.83 7.15 57.15 7.17 67.2 7.9
N1 59.83 57.15 67.2

Modifications of INCO 713-C Made to Improve Wrought Properties

2

TABLE

	Remarks							
	?en	1	1	ď	в	e	ď	45
	မ	1	0.10	1	!	:	}	ŀ
	ф	0.01	0.01	₹ 0.0	0.04	0.0	0.0	₹ •
	S1	0.14	0.14	0.14	0.14	0.14	0.14	0.14
/sis)	e.	06.1	98	1.97	1.94	1.94	1.92	1.91
ze Anal	II.	0.83	0.83	0.83	0.82	0.83	0.81	0.80
om Char	ပ	0.15	0.15	0.15	0.15	0.15	0.15	0.15
lated fr	Zr	0.18	8	0.18	0.18	90.0	0.08	0.08
lon (Calcu	ir Al W Mo Zr C T1 Fe	4.17	4.17	4.17	5.08	5.08	5.04	5.01
mpositi	3	!	i	1	;	:	0.97	1.93
cal Cc	A1	5.70	5.70	5.70	6.10	6.10	5.56	5.52
Chem	Cr.	12.70	12.70	12,70	12.49 (12.49	12.39	12.30
	Ta	2,15	2.15	2,15	2.60	5. 8	2.58	2.57
	N1	72.18	72.19	72.18	70.97	70.97	70.42	69.88
	WELT INCO 713-C Modifications						High Ta-Mo B-W Mod	
	MEL	547	N.	549	220	551	552	553

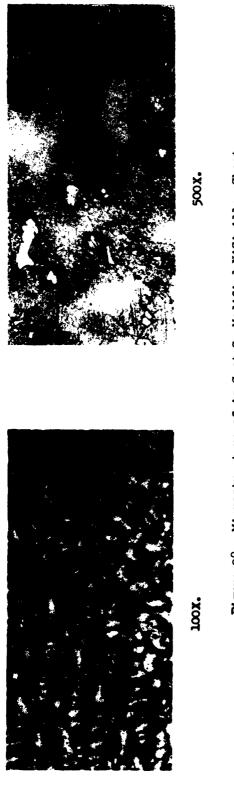


Figure 28. Microstructure of As-Cast Cr Modified NASA Alloy Sheet (17.68% Cr, Melt 426). Etch, No. 3.

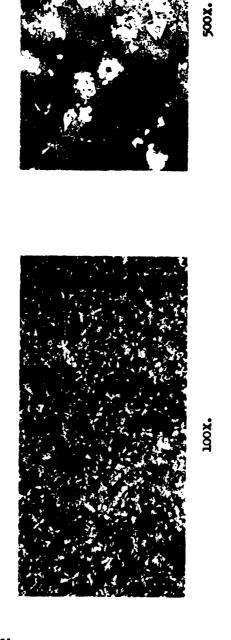
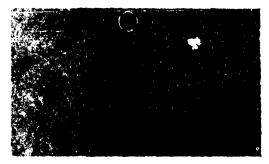
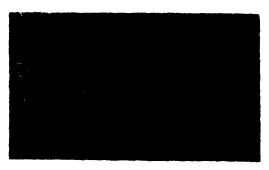


Figure 29. Microstructure of As-Cast Cr-Al Modified NASA Alloy (17.17% Cr, 8.56% Al, Melt 427). Etch No. 3.



Melt No. 529. NASA alloy, Less 1% Zr, Plus 3% Misch Metal +.03% FeB + 1% W.



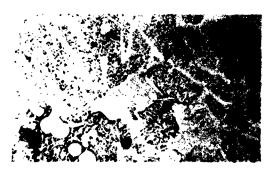
Melt No. 530. NASA alloy, Less 1% Zr, Plus .3% Misch + .03% FeB.



Melt No. 531. NASA alloy, +.88% Fe + +.030% FeB.



Melt No. 532. NASA alloy, +.030% FeB + 3% Ce + .88% Fe.



Melt No. 533 NASA alloy, Less 1% Zr +.09%C+.03 FeB+88% Fe+.3% Misch+1% W.



Melt No. 534. NASA alloy, +.09% C + .03 FeB + .88% Fe +.3% Misch.

Figure 30. Modified NASA Alloy As Cast (Melt 529, 530, 531, 532, 533, 534). Etch No. 4. 500X.



Macro-Etched, Rolled Inco 713c Showing That Cracking During Rolling Tends To Occur At The Grain Boundaries. 1.5x. Figure 31.

4. Effects of Melting Conditions on Alloy Composition

a. Tantalum Melt Losses

Table 6 lists 16 melts containing high percentages of tantalum and giving both charge analyses and chemical analyses showing the tantalum loss incurred for each alloy. All of these melts were made in magnesia crucibles. Although up to 26.52 percent of tantalum was added to these melts, the maximum tantalum retained in the castings was 11.06 percent. As a result of these tests, it would appear that no more than about 11% tantalum can be held in a nickel alloy melt made in a magnesia crucible. Any tantalum added beyond this equilibrium percentage apparently reacts with the magnesia in the crucible and is either vaporized as the oxide or forms a slag.

b. Magnesium Pickup

Table 7 lists four melts which had the magnesium content of the resultant castings determined by chemical analysis. All four melts were made in magnesia crucibles. Since magnesium metal has a boiling point below the melting temperatures of the nickel alloys, any magnesium metal found in the casting represents in effect a dissolved gas in the metal at the time of pouring. This residual magnesium content varied from 50 to 100 parts per million in the melts analyzed. It is interesting to note that the melts that are high in magnesium are also high in nitrogen content. Both magnesium and nitrogen contents would therefore appear to be dependent on the same factor; namely, the efficiency with which the gaseous constituents are removed from the metal by bubbling argon gas through the melt. The contents of both gases appear to be independent of alloy composition. The magnesium content observed does not appear to have any large adverse effect on the properties of the metal. Later melts made in zirconia crucibles would be expected to pick up some zirconium from the crucible. Since these alloys normally are specified to contain some zirconium, this would not be expected to have a detrimental effect. It would be preferable to have a crucible which did not react with the melt, since crucible-metal reactions would not appear to be the ideal way to make alloy additions.

c. Boron Pickup

Table 8 shows that the boron content in four melts of nickel base alloy made in magnesia crucible varies

from 14 ppm to 82 ppm. This boron might conceivably have come from one of the items of melting stock as an impurity. Some of this boron in the modified Inco 713c alloy almost certainly came as one of the alloying constituents of the Inco 713c base material melted. On the other hand, the magnesia crucibles contain from 1500 to 2000 ppm of boron (see Table 9). Some of the boron content probably came from a metal-crucible reaction. Again it would appear desirable to make whatever boron additions are desirable by a means other than crucible-metal reactions.

d. Nitrogen Content

Table 10 shows that the nitrogen content of four nickel alloy melts made in a magnesia crucible and bubbled with argon gas ranges from 37 ppm to 82 ppm. The nitrogen content appears to be independent of alloy composition and in particular independent of chromium content. It would appear to be a function of the effectiveness of the argon bubbling performed in the given melt. The effect of small amounts of nitrogen in the alloy would be expected to be analogous to that of carbon; notably, to decrease ductility and raise strength. In view of the small amount present in these melts, the nitrogen content does not appear likely to be harmful. If larger amounts of nitrogen were present, it might cause gas porosity in the castings. This was not a problem in castings made in this program. Specifically, the argon bubbling procedure used throughout this program was originally developed for the express purpose of eliminating gas porosity in castings and it has been successful in doing this.

B. Alloy Sheet Improvement by Rolling

1. Initial Rolling Trials at Metals and Controls

The original plan of operation for this contract called for all melting and casting operations to be performed at Chance Vought and for all rolling operations to be performed at Metals and Controls. The initial rolling trials were performed at Metals and Controls.

Most of the work at Metals and Controls was devoted to efforts to cold roll Inco 713c, NASA alloy, and Ta-C modified Inco 713c. Initial cold rolling trials showed that all three nickel alloys cracked severely after from 5-20% reduction. This cracking occurred at the edge and also as a multitude of small cracks distributed over the

७। TABLE

Melt Losses in High Tantalum Content Nickel Base Alloys Melted in Magnesia Crucibles

Change in	in Melting	9	5.59 \$.34		÷0.75	÷0.18	₹0.0-	-0.05	1	-0.23	9.1 0	-	-3.47		
	Mn Ta	8.02 7.75 16.26	0.02 10.87 8.00 8.00 8.34	0.02 11.06	5.77 8.00 8.10	8 8 8 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9		 8.8.	88.8	8.00 7.77		7.17	1	17.58	Magneslum Content 0.005 0.010
	S1 Cb 1	0.12	0.191.680.02	72	0.19 0.78	0.06	0.03	0.13	·····································	0.0	0.13	70.	0.15 1.38	0.161.07	cb 1.68
	N ₂ S	37 ppm	82ppm 60ppm	41ppm 0								11		-	Mn S1 0.12 0.02 0.19
	æ Ø	53ppm 0.005	(82ppm 0.010	200°0 mdd					11			11		{	N2 Mar 37 ppm 82ppm 0.0
	S B	<10ppm 53j	<pre><10ppm 82ppm 0.010 <10ppm 14ppm 0.010 <</pre>	<10ppm 73ppm	111									1	bles B 53ppm 82ppm
	Ti Fe	0.82 1.28	0.89 1.30 c	0.83 1.34	0.35 1.55							0.701.05	0.64 1.15	0.52 0.93	stanta Cructhies S S <10ppm 53 <10ppm 82
	ပ	1.00 0.13 - 1.07 0.12 - 0.08 1.08 0	0.07 1.84 0 1.00 0.13 - 0.49 0.12 -	0.16 0.29 0 0.07 1.76 0	0.72 1.16 0	00 0.116-	61.0	0.16	0.17	1.73	0.187	0.73 0.25 - 0.07 4.57 0	0.17 1.66 0 0.05 2.96 0	0.09 2.46 0	7 Allōys Malted in Magnesia. Zr C Ti Fe 1.07 0.12 0.12 <10 0.97 0.84 0.891.30 <10
os1t	V Zr	2.23	2.51	11	0.00	2007	9 9 9 5 6 5	36.0	20.0	200	1.83	1.57			7 Allōys Meld Zr C 1.07 0.12 0.07 0.84
	Mo	1.01 4.01 3.88 1.06 3.66	3.60 4.00 3.69 4.05	3.64 3.18	3.47		2,8 2,8 3,8 3,8 3,8 3,8 3,8					3.18	8	7.75 3.50	T A B L E Of NICKET WO V 34.06 1.80
	A1 W	7.01 6.03 4.92	. 98 5.36 - 100 6.99 4 167 6.03 3 171 4.29 -	.83 5.59 -	2.01 5.8400 7.00 4 7.08 4 7.08 4		6.03	350	386	500	4.56 8.73	7.15 8.56 3	4.99	.17 4.1617	ontent of M M M M M M M M M M M M M M M M M M
	N1 Cr	67.58 6.02 69.00 5.81 60.75 10.71	63.94 10. 67.36 6.06 69.14 5.67	64.08 10.8 53.09 9.2	67.54 12. 67.37 6.0 66.68 6.7	200	0 K-0	ο~o	o w	\sim \leftarrow	-	57.15 17. 49.58 8.7	52.76 9.57 39.90 17.5	3.87 17.	Magnesium Co r Al 5 5.81 6.0 87 10.98 5.3
	ALLOY N	Wod Wod	/13-C Mod	713-C Mod	7-51/					od NASA	1 Mod	-Mo Mod		-7	T. 7
	ALI	NASA 18 Ta-C	8 E	INCO 18 Ta-C	18	18 NASA 18	13	ASAN 18 NASA	13	5	zi.		6 2	6/2	N1 0.69.0
	H	385 charge analysis chemical analysis 388 charge analysis	chemical analysis 389 charge analysis chemical analysis 390 charge analysis	chemical analysis 392charge analysis	chemical analysis 412charge analysis chemical analysis	chemical analysis chemical analysis ulfcharge analysis chemical analysis	422 charge analysis chemical analysis	423charge analysis chemical analysis 424charge analysis	chemical analysis	chemical anslysis	chemical analysis	chemical analysis charge analysis	chemical analysis 429charge analysis	chemical analysi	MELT ALLOY 385 NASA 385 NASA 385 NASA 388 NASA MASA MASA MASA 99.94
	MELT	38.	38 38	39%	41%	7 7	7 5 7 5	י ל ה	. יל בי	; % ====================================	124	428	429		MELT 385

Boron Content 53ppm 82ppm 14ppm 73ppm	
Cb 1.68 1.72	
81.00.12 0.12 0.12 0.20	
8 0.05 0.02 0.02	
MG 0.005 0.010 0.010	
N2 37ppm 0 82ppm 0 60ppm 0 41ppm 0	
Content of Nickel Alloys Melted in Magnesia Crucibles Composition Cr Al W Mo V Zr C Ti Fe S 1 Composition 5.81 6.03 3.88 4.06 1.80 1.07 0.12 0.12 < 10ppm 37 0.95 5.36 3.60 0.07 0.84 0.89 1.30 < 10ppm 85.67 5.03 3.69 4.05 2.10 0.49 0.12 0.07 < 10ppm 66 0.83 5.55 3.64 0.16 0.29 0.83 1.34 < 10ppm 41	
Fe Cruc 1.30 < 1.34 <	
T1 T1 0.89 0.83	
1n Mg C 0.12 0.184 0.12	~ !
2 Zr 2 Zr 1.07 0.49 0.16	0/1
081110 V V 1.80	FABLE
A Composite A 13 A 1	TA
N1cke W 3.88 3.69	
A1 of	
on Conte	
Boron TR 7.75 5. 10.87 10.87 10.83 34 5.	
N. 00. 00. 00. 00. 00. 00. 00. 00. 00. 0	
o 713c o 713c	
d INCO	
ALLOY HASA Ta-C Mod NASA Ta-C Mod	
18 19 19 19 19 19 19 19 19 19 19 19 19 19	
E	

ωl TABLE

Refractory Materials Used in Making Crucibles for This Program

Vendor Recommended Max. USB Temp.	4,00 <i>0</i> ,6	4000°F	3400 °F
Composition (Typical)	92.34% ZrO ₂ +HfO ₂ 0.08% S102 5.56% CaO 0.08% MgO 0.07% Fe ₂ U ₃ 0.30% A12 ^O 3 1.05% T1O ₂	96.39% MgO 1.91% CaO 1.21% S102 0.23% Fee23 0.26% A1203 1500-2000 ppm Boron	93% MgO 0.9% A1203 3.4% S102 2.0% F020 0.2% F0203 0.1% A1kal1 1500-2000 ppm Boron
Grain Siz e	-8 mesh	-30 mesh and 9 -100 mesh 1 mixed in equal 1 quantities 0	-1 ⁴ mesh
Vendor Designation	RM24.C	Magnorite x	1152
Vendor	Zirconium Corp. of America	Norton Co.	Norton Co.
Type of Refractory	Z 1rcon1a	Magnesia	Magnesia

TABLE 10 Nitrogen Content of Nickel Alloy Melted in the Plasma Resistance Furnace

	Nitroger Cb Content	37ppm 8 82ppm 60ppm 2 41ppm	
	ಕ	1.72	
	31	0.10	
	£	0.02	
	Ř	0.005 0.010 0.05	
rurnace	m	53ppm 82ppm 14ppm 73ppm	
Lacance	Ø	<10ppm <10ppm <10ppm <10ppm	
L res	р., Ф	0.12 0.07 1.34	
LIBBIL	ŢŢ	0.83	
ייי	ပ	0.12 0.84 0.12 0.29	
7 7071	Zr	1.80 1.07 0.07 2.10 0.49 0.16	
ב ביי	>	2.10	
110 1	Ā	4 64 60 600 60 64 64	
2	3	3.88	
	A1	0 00 0 0 0 0 0 0 0 0 0 0	
	Cr.	5.81 10.98 5.67 10.83	
	Ta	7.75 10.87 8.34 11.06	
:	N1	69.14	
		INCO 713C INCO 713C	
	ALLOY	NASA Ta-C Mod NASA Ta-C Mod	
	MELT	000 800 000 800	

entire surface of the sheet (see Figure 32). Tables 11, 12 and 13 show the work hardening of these three alloys by cold rolling. Figures 33, 34, and 35 show the same work hardening data in chart form. Inco 713c work hardens from Rc 42 to Rc 52 with about 20% reduction; Ta-C modified Inco 713c work hardens from Rc 39 to Rc 51 in about 30% reduction, while the NASA alloy work hardens from Rc 43 to Rc 57 with about 35% reduction. After 20% reduction, the respective increases in hardness are 10 Rc points for Inco 713c, 11 points for Ta-C modified Inco 713c, and 12 points for NASA alloy.

Figures 36, 37, and 38 show comparisons of the microstructures of as cast, as rolled, and rolled and heat treated Inco 713c, Ta-C modified Inco 713c (Melt No. 388), and NASA alloy. It will be noted that the Inco 713c is the only one of the three alloys which changes microstructure to an appreciable extent as a result of cold working and annealing. Inco 713c recrystallizes in five minutes at 2150°F after cold working. Neither of the other alloys shows any recrystallization after even 70 to 100 minutes of heat treatment at the same temperature.

Subsequent efforts to roll NASA alloy sheet by repeated cold rolling with heat treatment of the metal between cold rolling trials were all unsuccessful in reducing the NASA alloy sheet below about 70 mils. In all cases, crowning of the sheet during rolling became a serious problem. Furthermore, the as cast sheet has some variations in thickness. After successive rolling passes these variations in thickness remained, even though the over-all thickness of the sheet has been reduced as much as 20%. Since crowning added greater thickness variations, the as rolled sheet had substantially greater variations in thickness than did the as cast sheet. In some cases, these thickness variations in as rolled sheet were as high as 40 mils on a 70 mil sheet. Metal rolled on the mill with 20 inch diameter rolls showed a greater tendency to crown than the metal rolled on the 7 inch diameter rolls. Crowning in both cases was excessive and resulted in the rapid destruction of the entire sheet by the propagation of edge cracks across the whole width of the sheet (see Figure 32).

NASA had reported somewhat higher ductility in the NASA alloy at liquid nitrogen temperatures (-320°F). Therefore an effort was made at Metals and Controls to roll NASA alloy chilled to liquid nitrogen temperature. This test resulted in the severe cracking at about the same reduction as sheet rolled at room temperature.

NASA alloy cast sheet was encapsulated in 300 series stainless steel and cold rolled at Metals and Controls. This resulted in the complete breakup of the NASA alloy sheet into small pieces (see Figure 39).

Another piece of NASA alloy sheet was encapsulated in 300 series stainless steel and rolled at 1500°F. This also resulted in the complete breakup of the NASA alloy sheet.

The relative location of metal preheating furnaces and rolling mills in the Metals and Controls plant was such that the maximum practical rolling temperature possible was 1500°F. Hot rolling of the NASA alloy at 1500°F was attempted at Metals and Controls, but this was unsuccessful in reducing the sheet beyond the point obtainable by cold rolling.

Because of the continued difficulties in rolling thin sheet on the conventional rolling equipment available at Metals and Controls, together with the limitation on the maximum practical rolling temperature, further rolling trials at Metals and Controls were suspended.

The detailed results of all of the rolling trials performed at Metals and Controls are included in Appendix F which is a summary of all the rolling trials made in this program.

2. Hot Rolling of Sheet on the Rigid Mill

The results of all the rolling trials done on the rigid mill are reported in Appendix F. The results of these tests can be summarized as follows:

- a. The optimum procedure for all nickel base alloys rolled in this program requires a reduction per pass in mill setting of 3 mils per pass. This reduction per pass applies to both the two high and the four high configurations of the rolling mill and to all gauges of materials being rolled down to a minimum of 20 mils. Below 20 mil sheet thickness, smaller reductions are taken.
- b. The roll speed used for most tests was 97.5 surface feet per minute.

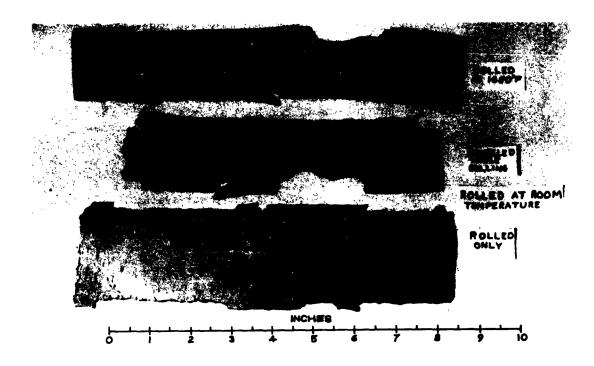


Figure 32. Typical Examples of NASA Alloy Rolled at M and C.

TABLE 11
WORK HARDENING TEST ON MELT 388 Ta-C MOD. INCO 713C

Pass No.	Thickness (.001")	Hardness $(^{R}_{\mathbf{c}})$	Pass No.	Thickness (.001")	Hardness (R _C)
0	109+1	39 - 1	7	79+2	51 <mark>+1</mark>
1	104-2	43 <u>+0</u>	8	75 +2	51 + 0
2	99 <u>*</u> ?	44 <u>+</u> 2	9	72 ⁺²	51 -2
3	96 ‡]	47 -0	70	69+2	51 <mark>+1</mark>
4	92 <mark>-2</mark>	49+0	11	65 +1	51 <u>+1</u>
5	87 +2 -1	50 +0	12	61 <mark>+1</mark>	52 ⁺⁰
6	83+2	50 +1	13	56 -1	52 <mark>-1</mark>

TABLE 12

WORK HARDENING TEST ON MELT 404 INCO 713C

Pass No.	Thickness (.001")	Hardness R _c
0	101+3	42 ⁺⁰
1	99-2	43 ⁺¹
2	96 <mark>+1</mark>	47 ⁺⁰
3	94-2	50 ⁺¹
4	89-1	50 + 0
5	85+2	51 <mark>+1</mark>
6	81-0	51 <mark>+1</mark>

TABLE 13
WORK HARDENING TEST ON Melt No. 425 NASA ALLOY

Pass No.	Thickness (.001")	Hardness R _C
0		
ı	100+2	43 ⁺⁰
2	97+1	47 ⁺¹ .5
3	96 <mark>+0</mark>	48-1
4	94 -1	50 -1 5
5	92 <mark>+1</mark>	50 <mark>+1</mark>
6	91 <mark>+1</mark>	51 <u>+2</u>
7	89 +1	52 <mark>+2</mark>
8	87 <u>+0</u>	54 + 2
9	85 + 0	54 ⁺³ -1.5
10	82 <mark>+1</mark>	54 ⁺¹
11	80 -1	55 + 2
12	76 -1	55 <mark>+1</mark>
13	74 <mark>+1</mark>	56 -1
14	70 -1	55 -1
15	65 -1	57 <mark>+2</mark>
16	62 <mark>+1</mark>	57 +. 5
17	58 ⁺¹	58 +2.5
18	55 ⁺⁰	57 +2.5

6 Rockwell C Hardness Heat No. 404 During Cold Rolling. Work Bardening Of Inco 713c,

Figure 33.

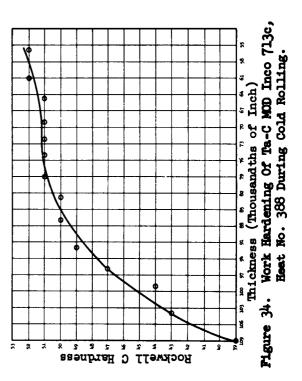
Thickness (Thousandths of Inch)

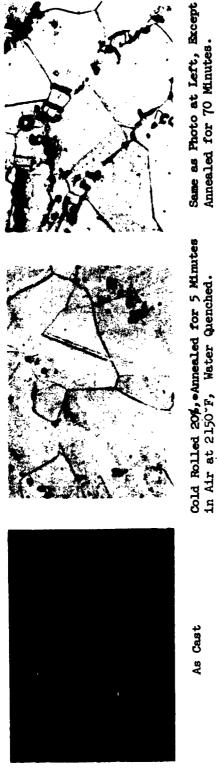
Rockwell C Hardening of Inch)

Thickness (Thousandths of Inch)

Figure 35. Work Hardening Of MASA Alloy, Heat

No. 425 During Cold Rolling.











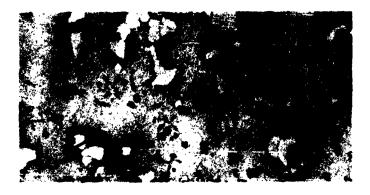
As Rolled + 5 Minutes Anneal.

As Cast.

As Rolled + 70 Minutes Anneal.

Effects of Cold Rolling (20%) and Annealing at 2000°F on the Microstructure of Ta-C Modified Inco 713c, Melt 388. Etch No. 3. 500X. Figure 37.

Figure 36.



As Cast.



Cold Rolled 14%, Annealed 10 Minutes in Air at 2150° F, Water Quenched.



Cold Rolled 14%, Annealed 100 Minutes in air at 2150°F, Water Quenched.

Figure 38. Effects of Cold Rolling and Annealing on the Microstructure of NASA Alloy, Melt 419. Etch No. 3. 500X.

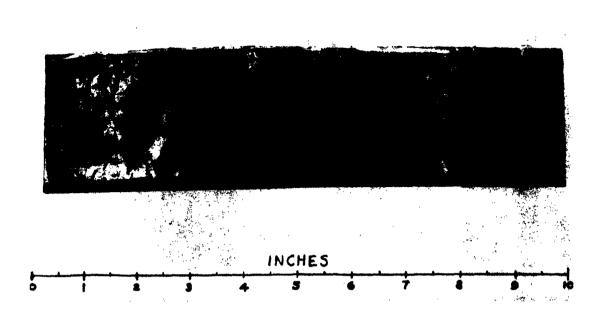


Figure 39. NASA Alloy Rolled in Stainless Steel

c. Optimum rolling temperatures for each alloy rolled are as follows:

Inco 713c 1950°F

nasa 1900°f

Ta-C modified Inco

713c (Melt No. 388) 2000°F

No. 429 Alloy 2100°F

- d. The two high mill configurations (6" diameter working rolls) should be used down to 40 mil sheet thickness.
- e. The four high configurations (1 1/2" diameter working rolls) should be used from 40 mil sheet thickness on down to foil gauges.
- f. The as cast sheet should not vary in thickness more than about 20 mils. The less the variation, the less likely it is that cracks will appear in the sheet during rolling.
- g. Fine as cast grain size metal rolls better than coarse grain metal.
- h. Inco 713c rolls better and with much less tendency to crack if the percentages of strengthening elements are kept on the high side of the specification, or if small percentages of tungsten are added.
- i. Shrinkage porosity which does not open to the outside will be sealed up during rolling, but shrinkage porosity of any kind will contribute to cracking of the sheet during rolling if other conditions are adverse.
- j. Surface pits and defects will not initiate cracking or failure during rolling but they may weaken the sheet during testing.
- k. Avoidance of crowning or curling of the sheet during rolling is vital if serious cracking is to be avoided.
- Prolonged solution heat treatment of the sheet either before rolling or after a few rolling passes causes rapid failure of the sheet by cracking in almost all instances. The reaon for this is not directly apparent.

m. Proper setting of the rolls prior to making the initial pass is important. Parallelism of the roll axes of rotation must be maintained within .001" to .003" at all times.

Refer to Figures 40 through 42 for typical examples of sheet rolled at Vought. Figure 43 shows the microstructure of hot rolled Inco 713c. NASA alloy and No. 429 alloy, all reduced 90%. Inco 713c exhibits a fine grained, typically hot rolled structure. NASA alloy has a heavily fibered structure typical of a cold rolled metal, even though it was rolled at 1825°F. The No. 429 alloy shows no evidence of either recrystallization or of cold rolling in its structure, even though it had been reduced in thickness 90%. Figure 44 shows the microstructure of Inco 713c foil cold rolled from hot rolled sheet. Figure 45 shows the change in microstructure in NASA allow with various reductions in thickness and with rolling at several temperatures. NASA alloy shows no appreciable change in microstructure in comparing as cast with 20% reduction. Further reductions to 50% and 68% at 1750°F and 2000°F show typically cold rolled structures with fibering becoming increasingly evident. Hot rolling at 2250°F to 68% reduction still shows considerable evidence of fibering, but it also shows some tendency toward relief and recrystallization. If higher rolling temperatures could be achieved, true hot rolling might occur, but this is prevented by the fact that the solidus temperature is only a little above 2250°F. Figure 46 shows a comparison of the structures of as cast No. 429 alloy with the same alloy in the hot rolled condition. Some of the carbide precipitates appear to be broken in the rolled structure but there is no evidence of fibering or of true recrystallization. Figure 47 shows a comparison of as cast and hot rolled Ta-C modified Inco 713c (Melt No. 388) microstructures. The carbide precipitate is apparently broken up into elongated groups of precipitates by rolling. There is no evidence of recrystallization in the rolled structure. Figures 48 and 49 show electron micrograph studies of the structures of hot rolled No. 429 and NASA alloys. The No. 429 alloy has a great deal more areas of massive precipitate than the NASA alloy although both have areas of massive precipitate. The structure in the No. 429 alloy is very difficult to resolve. It is clear from the microstructures that both alloys employ precipitation hardening as one of their strengthening mechanisms.

3. Analysis of Reasons for Failure of Rolling Triels on Conventional Mills

Efforts were made to roll NASA alloy, Inco 713c, and Ta-C modified Inco 713c on conventional rolling mills both at Metals and Controls and elsewhere. None of these efforts

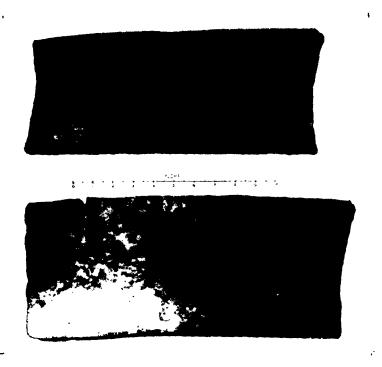


Figure 40. Rolled Inco 713c Sheet (Fine Grain).

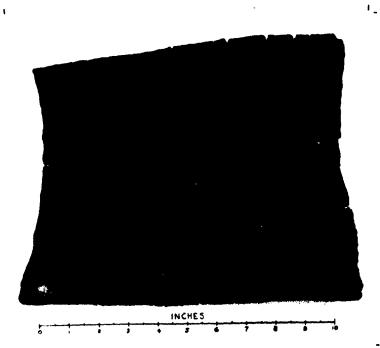


Figure 41. Rolled Inco 713c Sheet (Coarse Grain).



Figure 42. Inco 713c, Rolled at Vought .025" Thick.



713C, Melt 404, Reduced From As-Cast To.015", 90% Reduction At,2000°F, As Rolled.

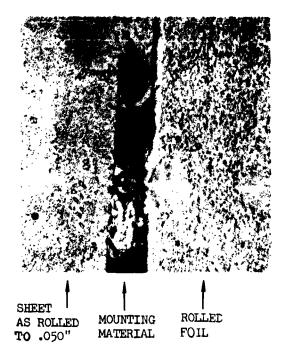


NASA Alloy, Melt 462, After Tensile Test At.050", Rolled To .015"(Total Reduction 90%) At 1825°F, As Rolled

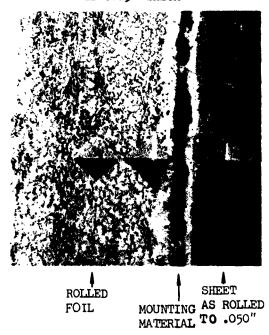


No. 429 Alloy, Melt 457, After Tensile Test At .050", Reduced To .015" (Total Reduction 90%) At 2100°F, As Rolled.

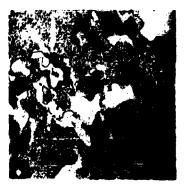
Figure 43. Comparison of the Effects of 90% Reduction In Thickness By Hot Rolling On Inco 713c; NASA, And No. 429 Alloys. Etch No. 4. 500X.



ROLL 234 DOUBLE PACK ROLLED .005" THICK



ROLL 235 TRIFLE PACK
ROLLED .0035" THICK
Figure 44. Inco 713c, Melt 550, as Rolled at Room Temperature, Etch
No. 4. 500X. (Note 200 gm Micro-Hardness Identations.)



NASA Alloy, Melt 462, Rolled 20% At 1500°F As Rolled.



NASA Alloy, Melt 462, Rolled 50% At 1750°F As Rolled.



NASA Alloy, Melt 462, Rolled 68% At 2000°F As Rolled.



NASA Alloy, Melt 462, Hot Rolled At 2250°F 68% As Rolled.

Figure 45. NASA Alloy, Hot Rolled To Various Reductions In Thickness At Several Temperatures. Etch No. 4. 500X.

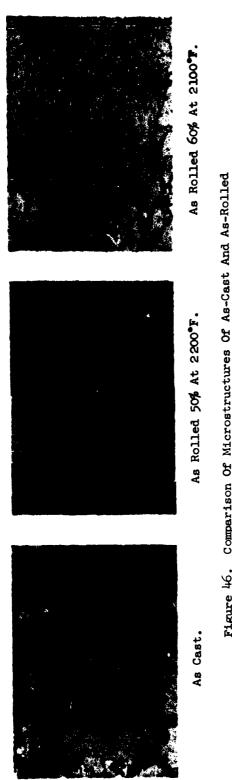


Figure 46. Comparison Of Microstructures Of As-Cast And As-Rolled No. 429 Alloy. Etch No. 4. 500X.

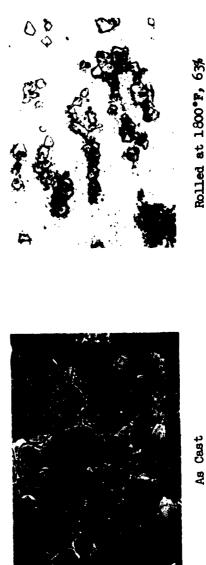


Figure 47. Comparison of Microstructures of As-Cast and As-Rolled Ta.c Modified (16.26% Ta, 1.08% C) Inco 713c, Melt 388. Etch No. 1. 500X.

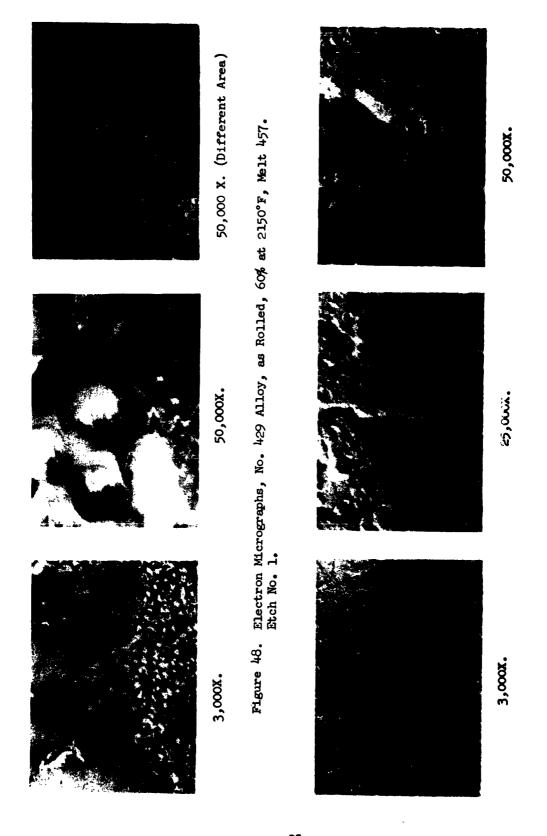


Figure 49. NASA Alloy, Rolled 60% at 2000°F, Melt 462.

succeeded in rolling these alloys into sheet having any substantial, uniform reduction in thickness from the as cast sheet. On the other hand, procedures were developed on the rigid rolling mill which permitted the hot rolling of 15 to 30 mil sheet from NASA alloy, Inco 713c, Ta-C modified Inco 713c, and No. 429 alloy. Experimental trials on the rigid mill indicate that hot rolled Inco 713c sheet could be cold rolled down to 3.5 mil foil in widths up to three inches. This foil was cold rolled from 20 mil sheet previously hot rolled on the rigid mill. Figure 50 shows four pieces of Inco 713c foil rolled on this mill. Once hot rolling procedures had been developed at Vought, an effort was made to use these procedures to hot roll NASA alloy sheet on a conventional rolling mill having the same size working rolls. Under these conditions, the conventional rolling mill produced a severely cracked sheet with 10 mil crown (see Figure 51).

It is believed that the primary reason for the failure of rolling trials on conventional mills is the presence of excessive crowning of the rolled material.

The ability of the rigid mill to roll alloy sheet which was not rollable on the conventional rolling mills used in this program is believed due to the unique design features of the Vought mill which minimize sheet crowning. This mill is designed to maintain a greater uniformity of gap between the working rolls with variations in rolling load than is possible with conventional design. A more detailed discussion of the design of this mill is given in Appendix B.

Crowning of the sheet during rolling is a direct result of variations in the gap between the working rolls caused by the separating force generated by the working of the metal sheet passing between the rolls. Crowning is the direct cause of sheet failure during rolling by the deep penetration of edge cracks. In rolling relatively brittle materials, a slight degree of edge cracking always occurs. This is caused by the fact that in the center of the sheet the total effect of diminishing the thickness of the sheet goes into elongating the sheet. At the very edge of the sheet, the effect of diminishing the thickness of the sheet by rolling goes partly into the elongation of the sheet and partly into displacement of the metal along the axis of the roll, thus widening the sheet somewhat. Since this is true, the sheet at its very edge does not elongate as much as the same sheet a little way in from the edges. Unless the metal has substantial tensile ductility, this soon results in small edge cracks appearing to relieve the tensile stress caused by this greater elongation of the main portion of the sheet. (See Figure 52). In the absence of crowning, these

edge cracks will normally penetrate about an eighth of an inch and then stop. If crowning is taking place, first one side of the sheet will be placed under abnormally high tensile stress by the elongation of the other side of the sheet during rolling; and then the process will be reversed as the cocking of the rolls reverses itself. As a result of these abnormally high tensile stresses, the edge cracks will rapidly penetrate to the center of the sheet, thus destroying the sheet (see Figure 53).

C. Alloy Sheet Improvement by Heat Treatment

1. Inco 713c

a. Recrystallization

Rolled Inco 713c sheet having as little as 20% reduction recrystallizes completely at 2150°F in five minutes. Figure 36, page 44, shows such a recrystallized structure. Figure 43, page 51, shows the microstructure of Inco 713c reduced 90% by rolling at 2000°F. This structure is also completely recrystallized. It is, therefore, evident that rolled Inco 713c sheet will rapidly recrystallize at 2000°F although a specific minimum time cannot be stated on the basis of available data.

b. Solution Heat Treatment

The object of solution heat treatment is not to recrystallize the alloy, although this may be an incidental result of the heat treatment. The object of solution heat treatment is to dissolve the carbide and other precipitates in the matrix alloy and redistribute these precipitate constituents by diffusion while they are in solution. After this redistribution has taken place, the alloy is cooled rapidly to retain in solution the precipitate forming constituents as an undercooled non-equilibrium structure. In this case, subsequent heating to intermediate temperatures will produce "aging" reactions where the precipitate will be formed under controlled conditions. If the solution heat treated alloy is permitted to cool more slowly, the precipitate forming constituents will precipitate out during cooling in less controlled manner. In general, it would be considered more desirable metallurgically to rapidly quench the solution heat treated metal and then form the precipitated phases through controlled "aging" heat treatment. In practice, very rapid quenching of metal structures will result in greater warpage of the structure than

slower cooling and may result in cracking of the metal due to thermal shock. As a result, the most useful cooling rate from solution heat treatment temperature is usually a compromise between the most desirable metallurgically and that most desirable for minimizing warpage and cracking. In this program, water quenching of NASA alloy from solution heat treatment temperature was found to frequently cause cracking of the alloy. Therefore, most of the quenching of NASA alloy in this program was done by air cooling. Air cooling was also used for the other alloys investigated throughout most of the program in order to preclude cracking problems, and in order to maintain a uniform procedure for all alloys.

Both macro and micro segregation of substantial degree occur of necessity in cast alloys of complex composition. These reasons are explained in detail in Appendix A. The only method possible for reducing segregation in a cast alloy is the use of prolonged solution heat treatments. The length of time at solution temperature required for an effective heat treatment depends on the diffusion rates of the elements involved and on the distance which the atoms in question must move by diffusion to achieve the desired end. In the instance of interstitial alloying elements, such as carbon, nitrogen and hydrogen, diffusion rates are quite high and relatively short solution heat treatment times are required to homogenize even badly segregated alloys. Where the segregated alloy constituents are substitutional elements of large atomic size. such as tungsten, tantalum, molybdenum, etc., diffusion rates are very low at the highest temperatures possible without melting the alloy. Hence, these alloys require long solution heat treatment times in order to homogenize the alloy. In normal alloys the grain boundaries form a continuous phase and the grains themselves a discontinuous phase. Therefore, the strength of the alloy structure as a whole is largely affected by the strength of the grain boundary metal composition. In a normal alloy casting, the grain boundary metal invariably has a lower melting point than the center of the grain. In castings of complex alloys, the grain boundary metal may have a melting point appreciably lower than that indicated by an equilibrium diagram for the alloy as a whole. It is therefore particularly important to minimize such grain boundary segregation in complex alloys designed for use near their melting points. This applies especially to the nickel base superalloys investigated in this program.

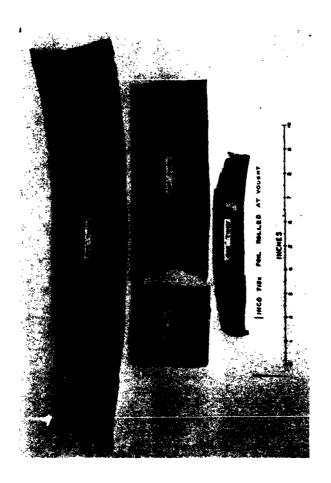


Figure 50. Inco 713c Foil Rolled at Vought. Melt No. 550.

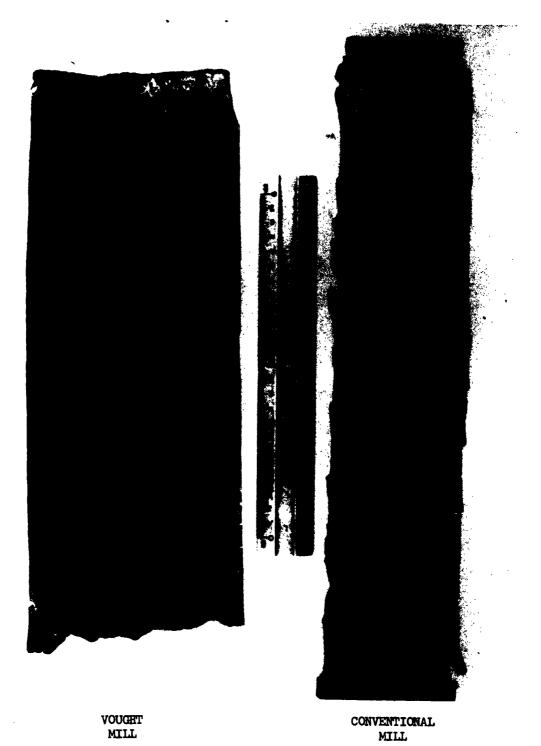


Figure 51. NASA Alloy Rolled At 1850°F.

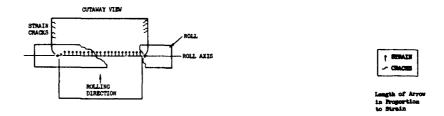


Figure 52. Diagram Showing Strain Distribution in Sheet Being Rolled on a Rigid Mill with Resultant Edge Cracks.

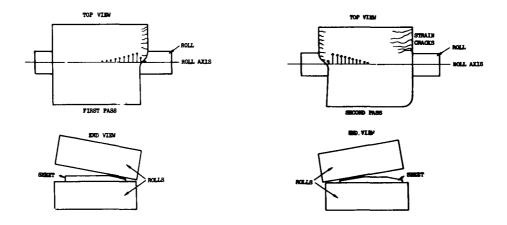


Figure 53. Diagram Showing Resultant Strain and Crack Distribution During Two Successive Passes on Conventional Mill.

Solution heat treatments of up to 24 hours at 2150 F indicate substantial improvement of both the room temperature and elevated temperature strength of cast Inco 713c. Table 14 shows a comparison of both room temperature and elevated temperature strength for ascast and heat treated Inco 713c. Figure 54 shows the homogenization of a dendritic, as cast, Inco 713c microstructure obtained by a 3 hour solution heat treatment at 2150°F. Figure 55 shows the progressive change in grain boundary conditions resulting from increasing times of solution heat treatment for cast Inco 713c. The precipitate in the grain boundaries was determined to be TiC by preferentially dissolving the matrix metal chemically and determining the crystal parameters of the precipitate by X-ray diffraction. It will be noted in Figure 55 that a 3 hour heat treatment has reduced the amount of precipitate at the grain boundary, but a 21 hour treatment has not only cleared the grain boundary of precipitate but has also changed the character of the grain boundary. The longer heat treatment corresponds to improved strength at both room and elevated temperature. Figure 56 shows TiC precipitate at the grain boundary of Inco 713c which has been fractured by a microhardness indenter. This indicates the brittleness of this precipitate at room temperature.

Figure 57 shows the microstructure of Inco 713c cast into a one-inch thick section and heat treated for 3 hours at 2150°F. The specimens of Inco 713c previously shown had been cast into sections one-half inch thick or less. It will be noted in Figure 57 that the degree of grain boundary segregation, even after 3 hours solution heat treatment, is still substantial when compared with the as cast structure of a thinner casting. It is apparent from this, that the heavier the section cast the more difficult it is to remedy grain boundary segregation by solution heat treatment.

Figure 58 shows electron micrograph studies of the effects of solution heat treatment at 2150°F on the TiC precipitate in the grain boundaries of cast Inco 713c. Figure 59 shows the effects of varying times of solution heat treatment of Inco 713c on its macroetching characteristics. The macroetched as cast Inco 713c shows a ridge of TiC precipitate remaining unattacked along the grain boundary with the depleted zone on each side of this ridge preferentially attacked by the etchant. This shows a marked tendency for preferential attack along the grain boundary by chemical etchants in the as cast condition. It is

reasonable to deduce from this that there would also be preferential attack in an analogous manner by oxidation at elevated temperature. The Inco 713c solution heat treated for 3 hours at 2150°F shows preferential attack along the grain boundaries, but it no longer shows a retained ridge of TiC at the grain boundaries. This evidence, coupled with the microstructures previously discussed, clearly indicates that the TiC precipitate has been largely redistributed out of the grain boundaries by the 3 hour solution heat treatment.

Carbon is an interstitial element having a high diffusion rate and titanium is a substitutional alloying element having a relatively small atomic diameter and hence would be expected to have a relatively high diffusion rate. It is therefore reasonable to believe that the TiC has diffused out of the grain boundaries, but that macrosegregation of other substitutional elements at the grain boundaries still exists in significant amount after 3 hours heat treatment. This would explain the continued preferential grain boundary attack after three hours heat treatment and the absence of the TiC ridge. After 21 hours solution heat treatment the preferential attack of the macroetching solution on the grain boundaries has completely disappeared. This would indicate that for this particular sample of cast Inco 713c, a solution heat treatment time of 21 hours at 2150°F is adequate to essentially homogenize the structure. It should be pointed out that this solution heat treatment might not be adequate for cast Inco 713c having coarser grain size or a greater amount of macrosegregation. These tests do indicate that a prolonged solution heat treatment time is required to homogenize complex nickel base alloys. These tests also provide some indication that solution heat treatment might minimize the tendency for preferential grain boundary attack by oxidation at elevated temperatures.

Table 15 shows the effects of long time solution heat treatments on the room temperature and elevated temperature tensile properties of rolled Inco 713c sheet. The most marked improvement in properties obtained by extending solution heat treatment time beyond 24 hours is in room temperature ductility. Solution heat treatment for at least 24 hours appears to be necessary to attain the best combination of elevated temperature tensile strength and room temperature ductility.

c. Aging Heat Treatments

The solution heat treated Inco 713c rolled sheet essentially met the target properties of the program so relatively little work was done on investigating aging heat treatments for this alloy. Only two aging temperatures were used; 1600°F and 1900°F. The effects of these aging heat treatments on solution heat treated rolled Inco 713c sheet are given in Table 16. One-half hour aging at 1900°F has had no appreciable effect on tensile properties at 1900°F. Aging heat treatments at 1600°F tend to improve elevated temperature ductility, but they tend to lower room temperature ductility. Aging at 1600 F does not appear to appreciably affect tensile strength. The aging heat treatments used in this program on Inco 713c do not appear to have had any appreciable effect on tensile properties other than to provide some improvement in elevated temperature ductility.

2. NAS. TaZ8 Alloy

a. Recrystallization

The NASA alloy is much more difficult to recrystallize than Inco 713c. Rapid recrystallization of the NASA alloy does not take place until a temperature of 2200°F is reached. Furthermore, even at this temperature recrystallization has been observed only on sheet which has received reductions in thickness by rolling of approximately 50%. Figure 38 on page 45 shows that heat treatment at 2150°F does not recrystallize NASA alloy cold rolled 14%. Figure 45 on page 53 shows that NASA alloy hot rolled at 2250°F has not recrystallized. Figure 60 shows that NASA alloy rolled to 45% reduction and then solution heat treated for 1/2 hour at 2200°F has undergone some recrystallization, although recrystallization does not appear complete. Figure 61 shows a comparison of the microstructure of as cast, as rolled, and recrystallized NASA alloy as shown by electron micrograph studies. The rolled and recrystallized NASA alloy had been hot rolled at 1650°F to 60% reduction and heat treated at 2200°F for 1/2 hour. Recrystallization appears complete, but the basic structure of the rolled and recrystallized alloy is substantially different from that of the as cast alloy. Since the 1900°F strength of the as cast alloy is appreciably greater than that of the best rolled and heat treated NASA alloy, it would appear that the as cast structure is to be preferred for high temperature strength.

TABLE 14

COMPARISON OF ROOM AND ELEVATED TEMPERATURE STRENCTH

ខំ

AS CAST AND HEAT TREATED INCO 713C

Ne lt	Condition	lest Temperature	Strength (ps1) Room Temperature	Strength (psi) Strength (psi) Room Temperature Temperature	Percent Room Temperature	Stress	Life Klongat bours) (\$)	Flongation (4)
	As Cast	Коош	118,100	126,300	2.5			
	As Cast + (A)	Room	122,100	130,600	3.0			
	As Cast	1700				30,000	10.8	0.4
	As Cast + (A)	1700				30,000	26.1	3.0
	As Cast + (B)	1700				30,000	15.7	•
67(c)	As Cast	Room	94,500	110,600	8.0			
67(c)	As Cast + (A)	Room	119,000	142,900	0.6			
67 (c)	As Cast	1700				30,000	20.5	96.0
67(c)	As Cast + (A)	1700				30,000	ш.2	12.0
102(C)	As Cast	Room	109,800	121,900	5.0			
	As Cast + (A)	Room	118,400	131,900	0.4			
	As Cast + (D)	Room	115,500	122,500	2.0			
	As Cast + (E)	Room	127,000	131,800	5.0			
	As Cast	1700				30,000	20.8	0.4
	As Cast + (A)	1700				30,000	3.5	5.0
	As Cast + (D)	1700				30,000	16.3	2.0
	As Cast + (B)	1700				30,000	१ हुन	3.0

H.T. 3 hours at 2150°F air cool
H.T. 3 hours at 2150°F + 24 hours at 1550°F
H.T. 3 hours at 1950°F Air Cool
H.T. 24 hours at 2150°F Air Cool

38088

⁶⁵

TABLE 15

Effect of Solution Heat Treatment on Rolled INCO 713C Sheet

Klongation	ROOM TEMPERATURE 1900°F	0.0 W.0	0.0	0.01		18.0
	1900°F	4,000 44,000	50,70	, 3, 98, 98,	38,200	51,400
Ultimate Tensile	ROOM TEMPERATURE			178,200	164,200	187,300
Yield	ROOM TEMPERATURE			143,900	15¢,600	135, 700
Test	a ma saladinar	1900 %	1900 F	ROCM & 1900 F	PROOM & 1900 F	ROCM & 1900 *F
Heat Treatment		as rolled hi hrs at 2150°F A.C.	= = = = = = = = = = = = = = = = = = = =	3 hrs at 2200 F A.C.	3 hrs at 2200°F, 170°F	oil quench 24 hrs at 2200°F A.C. R(
Roll	Name of the last	OIS	516	27	23	8 <u>1</u> 5
Melt		84.2	, <u>%</u>	† 0 1	₹ 04	101

TABLE 16

Effect of Aging Heat Treatments on Rolled and Solution Heat Treated INCO 713C Sheet

Elongation (\$)	1900*F	3.0	0.01	0*9	9.0	0.51
Ultimate Tensile Strength	1900 * F	000,444	50,800	20,000	47,800	oon 16th
Test Temperature		1900	1900	1900	1900	1900
Heat Treatment		as rolled	41 hrs at 2150°F + 24 hrs at 1600°F	41 hrs at 2150°F + 61 hrs at 1600°F	40 hrs at 2150°F + 24 hrs at 1600°F +1/2 hr at 1900°F	1/2 hr at 2200°F + 24 hrs at 1500°F
Roll			210	216	211	
Melt			548	550	742	



As Cast



Solution Heat Treated at 2150°F for 3 Hours and Air Cooled.

Figure 54. Homogenization of As-Cast Dendritic Structure in Inco 713c by Heat Treatment at 2150°F. Etch No. 1. 100X.



As Cast, Melt 56.



Heat No. 56, Solution Heat Treated At 2150°F For 3 Hours, Air Cooled.



Heat No. 104, Solution Heat Treated At 2150°F For 21 Hours, Air Cooled.

Figure 55. Effect Of Solution Heat Treatment Of Cast Inco 713c. Etch No. 2. 500X



Figure 56. TiC Precipitate in Grain
Boundary of As-Cast Inco 713c.
Note: Micro-Hardness Indentor
Shattered the Precipitate,
Indicating its Brittle Nature.
Etch No. 1. 1,000X.

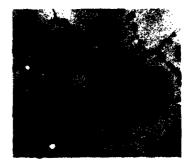
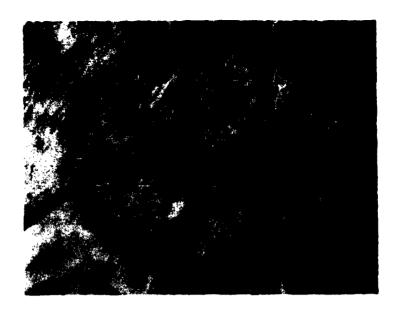


Figure 57. Inco 713c Section Cut from One-Inch Thick Specimen Heat Treated at 2150°F for 3 Hours. Etch No. 1. 100X.



As Cast

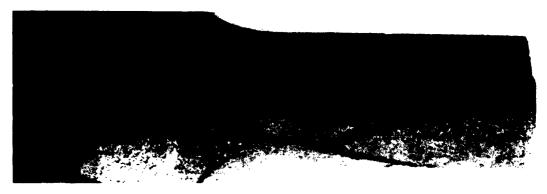


Solution Heat Treatment at 2150°F for 3 Hours, Air Cooled.

Figure 58. Effect of Solution Heat Treatment on Grain Boundary in Cast Inco 713c, Melt 67. Etch No. 2. 4,000X.



As Cast. 6X.



3 Hours At 2150°F, Air Cool. 6X.

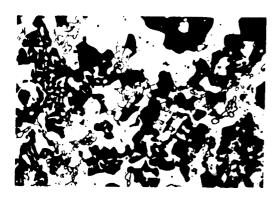


21 Hours At 2150°F, Air Cool. 4X.

3 Hours At 2150°F, Air Cool. 4X.

As Cast. 4X.

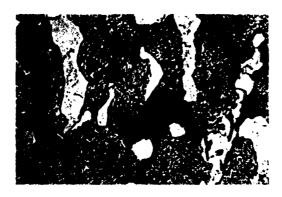
Figure 59. Comparison Of Macro-Etching Characteristics Of As-Cast And Heat-Treated Inco 713c.



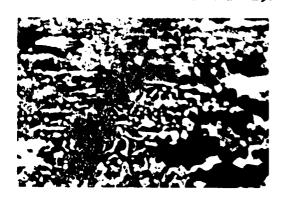
NASA Alloy, Melt 462, Reduced 45% at 1650°F as Rolled.



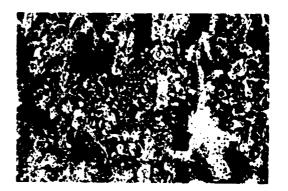
NASA Alloy, Melt 462, Reduced 45% at 1650°F, then Heat Treated 1/2 Hour at 2200°F, Air Cooled.



NASA Alloy, Melt 462, Reduced 60% at 1750°F, then Heat Treated 63 Hours at 1500°F, Air Cooled.



NASA Alloy, Melt 418, Reduced 50% at 1825°F, then Heat Treated 1/2 Hour at 2200°F, Air Cooled.



Same as Photo at Left, with Additional Heat Treatment of 24 Hours at 1600°F.

Figure 60. Effects of Heat Treatment on the Microstructure of Hot Rolled NASA Alloy. Etch No. 4. 500X.

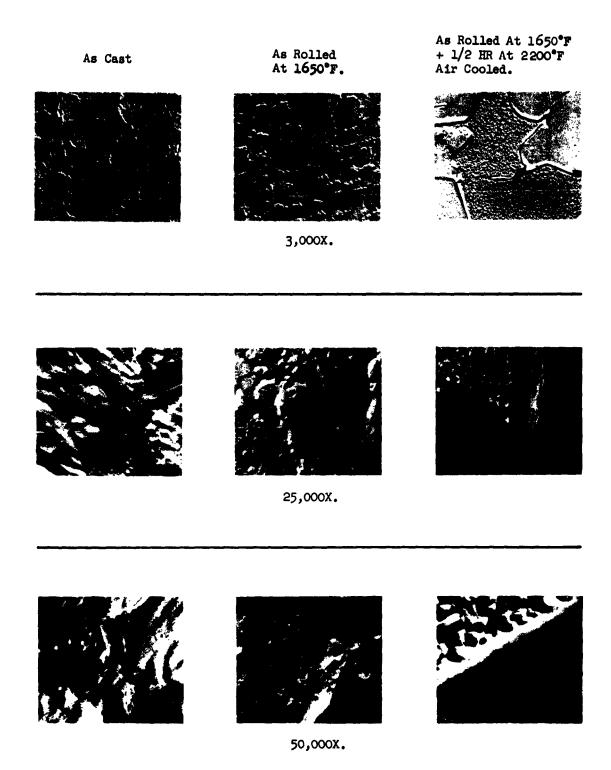


Figure 61. Comparison Of Microstructure Of NASA Alloy In The As-Cast, As-Rolled, And As-Rolled + Heat-Treated Condition.

In summation, rolled NASA alloy will recrystallize completely in 1/2 hour at 2200°F provided it has had 60% reduction in thickness by rolling before heat treatment. With 45% reduction in thickness, recrystallization is partial after 1/2 hour at 2200°F. Lesser reductions or lesser heat treatment temperatures do not result in appreciable recrystallization.

b. Solution Heat Treatment

Prolonged solution heat treatments do not materially affect the microstructure of NASA alloy as seen in the optical microscope. (See Figure 62). Figure 63 shows the effects on the as cast NASA alloy structure of 2000°F and 2200°F solution heat treatments as shown in electron micrograph studies. These solution heat treatments apparently cause the massive precipitate to breakup into finer form, and the higher the heat treatment the more complete the breakup. Table 17 gives a comparison of both room temperature and 1900°F tensile strengths of NASA alloy in the as-cast condition and after two different solution heat treatments. It will be noted that solution heat treatment increases room temperature tensile strength somewhat, but otherwise has no significant effect on the mechanical properties tested. A comparison of Figure 64 with Figure 65 shows that the variation in microstructure of NASA alloy caused by solution heat treatment is relatively small compared with the variation in microstructure of the same alloy from one part of a given casting to another part of the same casting. These differences in as cast structure are apparently due to differences in cooling rate of one part of the casting relative to other parts of the same castings.

Table 18 shows the effects of various solution heat treatment times on the mechanical properties of NASA alloy. The most noticeable improvement in properties resulting from prolonged solution heat treatments is a small increase in room temperature ductility. Tensile strength at 1900°F appears to decrease somewhat with longer solution heat treatment times, although this may be due to oxidation attack on the surface of the metal caused by impurities in the atmosphere used in heat treatment. At best, there is no significant improvement in 1900°F tensile strength as a result of long time solution heat treatment. Since the solidus point of the NASA alloy has been determined to be about 2300°F, and since temperatures appreciably below 2200°F

will not recrystallize the rolled structure, it would appear unlikely that any practical solution heat treatment can be devised to substantially improve the properties of the NASA alloy.

c. Aging Heat Treatment

Table 19 shows the effects of various aging heat treatments on solution heat treated, rolled NASA alloy. Aging heat treatments after solution heat treatment do substantially improve the mechanical properties of the NASA alloy. The aging treatment giving the best room temperature properties is 24 hours at 1750°F. This heat treatment gives somewhat lower room temperature tensile strength than some others, but most important it is the only heat treatment found to give usable room temperature ductility to rolled NASA alloy sheet. This heat treatment gives 4% to 5% elongation at room temperature, which is probably a practical minimum allowable figure if the alloy is to find practical use in rolled sheet form. The best 1900°F tensile properties were obtained with an aging heat treatment of 24 hours at 1600°F. It is likely that an intermediate aging temperature might result in an optimum balance of properties.

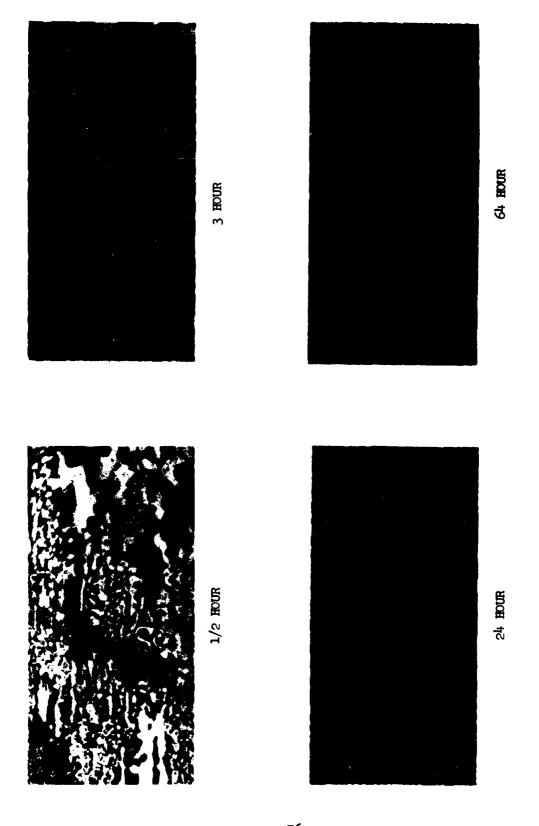
Figure 66 shows the results of an electron microscope study of rolled NASA alloy which was aged at 1500°F for 63 hours. This study shows that aging at this temperature without prior solution heat treatment does not materially change the structure from that existing in the as rolled condition.

3. No. 429 Alloy

Only a very limited amount of work has been done on the rolling and heat treatment of No. 429 alloy. The microstructure of No. 429 alloy is complex and difficult to adequately resolve due to the greatly varying rate of attack of standard etching solutions on the various microconstituents. No. 429 alloy has been hot rolled at 2100°F and at 2200°F. None of the alloy rolled has received subsequent heat treatment. There is more variation to be observed in the microstructure of a single casting of the alloy than there is variation between the microstructures of the alloy in the as cast and the hot rolled conditions. Only one tensile test was run on sheet rolled from this alloy, therefore no conclusions can be drawn as to the strength of No. 429 in the as rolled condition. (Text continued on Page 80.)

TABLE 17 Effect of Solution Heat Treatment on Cast NASA Alloy Sheet

Welt		Heat Treatment	Test Temperature	Meld Strength	Ultimate Tensile Strength	nsile h	KLOI	Klongation (\$)
				ROOM TEMPERATURE	ROOM TIGNIFIERA TURE	1900°F	ROOM TISAPERATURE	1900•1
386 and 394 393 394	₹6	as cast 16 hrs at 2000°F W.Q. 16 hrs at 2200°F W.Q.	ROOM & 1900 °F ROOM & 1900 °F ROOM & 1900 °F	120,900	121,100 92,600 137,600	53,200 52,300 53,300	0000	8.0 1.0
i		Effect of Solu	TABLE 18 Effect of Solution Heat Treatment on Rolled NASA Alloy Sheet	18 it on Rolled MASA	Alloy Sheet			
Melt	Roll	Meat Treatment	Test Temerature	Yield Strength	Ultimate The Strength	Ultimate Tensile Strength	Elongation (≰)	tion
				ROOM TIEMP	ROOM,TEEMP	1900°F	ROOM TEMP	1900°F
418 425,385	601 621 621 621	1/2 hr at 2200°F A.C. 3 hrs at 2200°F A.C. 3 hrs at 2200°F A.C.	1900 F HOOM & 1900 F HOOM & 1900 F	153,600	128,400 128,300	8,42,8 8,43,8 8,	0000	, 1, 8, 6, 6, 6, 6, 6, 6, 6, 6, 6, 6, 6, 6, 6,
422,302	ट्य 'हम	off quench	2 000 a mon	238 600	34.500	200		
125 385 162,418	ह्य इस १८० १५०	24 hrs at 2200°F A.C. 64 hrs at 2200°F A.C. 64 hrs at 2200°F, retort	HOOM & 1900 F HOOM HOOM & 1900 F	134,400	14,800 161,700	000,04	000	9 9
425,385	53,125	cooled 3 hrs at 2250°F A.C.	ROOM & 1900 °F		136,500	36,500	0.0	1.0
, -	Roll Winder	Effect of Soluti	TABLE 19 Effect of Solution Heat Treatment and Aging on Rolled NASA Alloy Sheet Treatment Triald Ultimate Ten Treatment Strength	end Aging on Rol Yield Strength	led MASA Alloy Sheet Ultimate Tenaile Strenath	et Tensile Eb	Elonge	10n (\$)
n Talk	Mannoe	as rolled	1900°F	ROOM TIEMP	ROOM TIEMP	1900°F	ROCM TISMP 1900 P	5.0001 5.0
424,425	150,199	64 hrs @ 2150°F + 64 hrs @ 1450°F	Room & 1900°F	207,000 (A)	217,000 (A)	50,600 (A)	0.0	2.0
424,418	143,148	61 hrs @ 2150 °F + 24 hrs @ 1600 F	Room & 1900°F	189,400 (A)	226,600 (A)	57,700 (A)	5.0	4.5
387,385	154,155	61 hrs @ 2150 °F + 24 hrs @ 1750°F	Room & 1900°F	135,200 (A)	197,400 (A)	49,500 (A)	5.0	2.0
385,385	152,152	61 hrs @ 2150°F + 64 hrs @ 1950°F	Room & 1900	128,300 (A)	175,200 (A)	48,700 (A)	3.0	1.0
		1/2 hr @ 2200°F + 24 hrs @ 1600°F	1900°F			43,200		14.0
424,415	126,114	64 hrs @ 2200 °F + 24 hrs @ 1450 °F	Коош & 1900°F		121,000	33,200	0°8	
387,419	171,911	64 hrs @ 2200 F + 64 hrs & 1600 F	Room & 1900°F	123,500	131,500	40,100	1.0	12.0
462,419	171,051	as before + 2 1/2 hrs @ 2000°F	Room & 1900°F	120,200	134,400	38,900	1.0	16. 0
81. ⁴	741	64 hrs @ 2200°F+ 24 hrs @ 1675°F	1900°F			41,900	3.0	,
425	221	64 hrs @ 2200°F+	Room & 1900°F	123,300	144,900	34,300		0.01
(A) Bec	cause of some (Because of some oxidation depletion and scaling during heat treatment, thickness of specimen was measured with pointed micrometer.	ng during heat tre	stment, thickness	of specimen was	messured with p	cointed micr	meter.



Effect of Various Prolonged Solution Heat Treatments on Rolled NASA Alloy at 2200°F, Air Cooled. Etch No. 4. 500X. Figure 62.



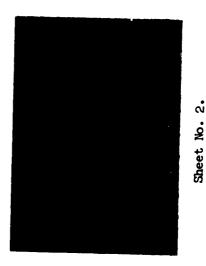
Heated to 2000 F for 16 Hours and Water Quenched. 25,000X.



Heated to 2200°F for 16 Hours and Water Quenched. 25,000X.





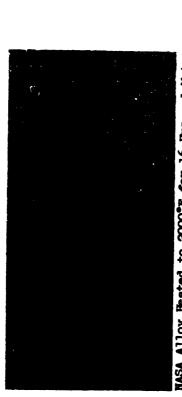


Sheet No. 4.



Heavy Bar Section.

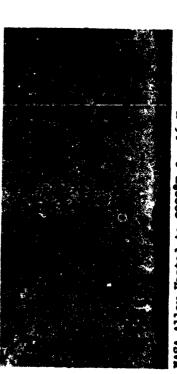
Variation of Microstructure within a Single Casting of NASA Alloy, Melt 385 as Cast. Etch No. 1. 100X. Figure 64.



MASA Alloy Heated to 2000°F for 16 Hrs and Water Quenched Heat No. 385 100X FeCl₃ + HCl Etch.



NASA Alloy Heated to 2200°F for 16 Ars and Water Quenched Heat No. 385 100X FeCl₃ + HCl Etch.



MASA Alloy Heated to 2000°F for 16 Hrs and Water quenched Heat No. 386 100X FeCl₃ + HCl Etch.



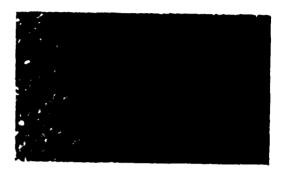
MASA Alloy Heated to 2200°F for 16 Hrs and Water Quenched Heat No. 386 100X FeCl₃ + HCl Etch.

Figure 65. Optical Microscope Study of the Effects of Two Different Best Trestments on Cast NASA Alloy.



3,000x.





25,000X.





50,000X.

Figure 66. Electron Micrographs Of NASA Alloy Hot Rolled 60%, Then Aged At 1500°F, For 63 Hours. Etch No. 1

4. Ta-C Modified Inco 713c (Melt No. 388)

Figure 37 shows that heat treating rolled Ta-C modified Inco 713c at 2000°F does not result in recrystallization of the alloy. This alloy therefore has a higher recrystallization temperature than Inco 713c. Work on this alloy was stopped relatively early in the program to provide more time for work on Inco 713c and NASA alloy, since the latter two alloys had better 1900°F tensile strength in the as cast condition. Hence, the recrystallization temperature of this alloy is not known, and the effects of any such recrystallization heat treatment are not known.

D. Rolled Alloy Sheet Evaluation

1. Tensile Properties

a. Inco 713c Rolled Sheet

Rolled and heat treated Inco 713c sheet has a tensile strength of approximately 50,000 psi at 1900°F and approximately 170,000 psi at room temperature. The ductility of this alloy ranges from 12% to 20% elongation at room temperature depending on heat treatment and from 6% to 10% at 1900°F depending on heat treatment. Figure 67 shows a plot of tensile strength and elongation versus test temperature for the range from room temperature to 2300°F. Table 21 gives a summary of the tensile strengths of rolled and heat treated Inco 713c in various conditions.

The rate of straining of the test specimen has a substantial effect on the tensile strength reported for nickel base alloys in the temperature range approximating 1900°F. Standard elevated temperature tensile test procedures generally call for a strain rate of 0.005 inches per inch per minute during the elastic portion of the test and from 0.05 to 0.1 inches per minute of head travel thereafter. The tests run at elevated temperature throughout most of this program were run at these or somewhat lower strain rates. Since only ultimate tensile strength and elongation were required, an extensometer was not used in making these tests. Therefore, direct control of strain rate was not possible. As is explained in Appendix C, strain rate was indirectly controlled by controlling load rate. The load rate used in this program results in a rate of straining less than standard rather than greater than standard. In order to determine the actual effect of straining rate during testing on the

reported tensile strength of Inco 713c, tests were run on comparable Inco 713c test bars at three substantially different rates of loading. Table 22 summarized the results of these tests and Figure 68 shows a plot of reported tensile strength versus calculated average strain rate. It has been determined by experiment that the total head travel of the machine during a test is approximately twice the actual strain in the gauge length of a standard 1" gauge length test bar used in this program. Figure 69 gives a plot of reported tensile strength versus time spent under load in the given test. It will be seen from these tables and charts that the true standard tensile strength of Inco 713c at 1900°F would be slightly higher than the results of the tensile test run in this program would indicate. This discrepancy is small and is not thought to be significant. It will be noted that large variations in strain rate will produce a variation of almost 300% in the reported value for the tensile strength of this alloy at 1900°F.

Table 23 shows the effect of strain rate on NASA alloy tensile strength at 1900°F. The data in Tables 22 and 23 therefore show that strain rate during testing substantially affects the reported tensile strength for both Inco 713c and NASA alloy.

b. NASA TaZ8 Alloy Rolled Sheet

NASA TaZ8 alloy sheet which has been rolled, solution heat treated at 2200°F and then aged at 1600°F has a 1900°F tensile strength of about 50,000 psi with an elongation of about 4%. The same alloy sheet will have a room temperature strength of about 170,000 psi and 4% to 5% elongation if it is aged at 1750°F instead of 1600°F. The tensile data on which these conclusions are based is given in Table 19 on page 75. No tensile data was obtained on rolled sheet for any temperature other than room temperature and 1900°F.

c. Other Alloys

A few isolated tensile tests were run on sheet rolled from Ta-C modified Inco 713c (Melt No. 388) but these tests are inadequate in number and in quality to use as the basis for any conclusions. No tensile tests were run on any other of the alloys rolled.

THE SO LENSILE DATA ON NICKEL ALLOYS

_
1900°F
and
room
Than
Other
Temperatures,
(Various

ELONGATION (%)	3.0	!	~ w w w	0 1 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
ULTIMATE TENSILE STRENOTH (PSI)	27,900 7,900	8,600	19,000 6,700 418 156,100	128, 200 108, 600 108, 600 128, 100 73, 200 86, 400 86, 400 11, 100 7, 300 5, 300
ULTIMATE TENSILE LOAD (#)	760 188	198	534 176 11	1788 1346 1346 1066 1066 1066 1168 860 860 860 860 860
AREA square inches	.02627 .02384	.02302	.02805 .02646 .02628	01395 01783 00124 00124 01713 01713 01184 01943 01943 01943 01943
THICK- NESS (0.001")	107.2 96.4	94.1	116.3 108.8 108.2	9000 9000 9000 9000 9000 9000 9000 900
TEST TEMPER- ATURE (°F)	2000	2100		1500 1500 1500 1500 2000 2000 2100 2200
CONDITION	as cast as cast	as cast	as cast as cast as cast	rolled + a.
MELT NUMBER	393 394	392	457 457 457	\$
ALLOY AND ROLL NUMBER	NASA	2(Ta-C) Modified INCO 713C	Ta-C-Cr-W Modified INCO 713C	INCO 7135, 201 INCO 7136, 201 INCO 7136, 201 INCO 7136, 201 INCO 7136, 230 INCO 7136, 231 INCO 7136, 232 INCO 7136, 230

(a) H.T. 40 hrs at 2150°F, +16 HRS at 1600°F.

TABL: 21 Tensile Properties of Rolled and Heat Treated INCO 713c

Remarks		Tield Street	Ultimate Tensile Strength (PSI)	Tensile (PSI)	Elo	Elongation (%)
		Room Temperature	Room Temperature	1900°F	Room Temperature	re 1900°F
as rolled	minimum average			41,800 42,900		0.0
rolled + HT 24 hrs at 2200 P, A. C.	minimum average	121,900	170,400 182,600	51,400 (single test)	12.0 est) 15.0	10.0
rolled + HT 3 hrs @ 2200°F, A. C.	minimum average		178,200 (single test)	28,800 35,800	12.0	0.0 8.0
rolled + HT 3 hrs @ 2200°F, 170°F oil quench (single test)	(single test)	124,600	164,200	38,200	14.0	0.9
rolled + Hr 1/2 hr @ 2200°F A. C.	(single test)			45,300		0•4
same as above + 24 hrs @ 1600°F A. C.	(single test)			004,64		0.51
rolled + HT 64 hrs @ 2200% A. C.	minimum average	122,600 123,300	170,200 172,800		18.0 19.0	
rolled + HT 41 hrs @ 2150°F A. C.	minimum average			41,900 46,400		2.0 6.5
as above + 24 hrs @ 1600° F	minimum average			6,500 50,200		0.01 0.01
as above + additional 37 hrs @ 1600°F	minimum average			45,300 47,700		6.0
rolled + Hr 40 to 60 hrs @ 2150°F + 24 hrs	minimum	141,200	156,100	45,700	3.0	8.0
at 1600%	average	(single test)		009*94		0.6

TABLE 22

Variation in 1900°F Tensile Strength of INCO 713c with Varying Strain Rate (All specimens rolled and HT 40 hrs @ 2150°F + 24 hrs at 1600°F)

Elongation	0.21	0°9	13.0	8.0	0°6	7.0	8.0	15.0
Ultimate Tensile Strength	30,700	31,500	24,700	946,400	47,800	(8) 000 (9)	65,300	69,700
Average Strain Rate Inches/Inch/Minite	0.0083	0,0083	210.0	0.021 (A)	0.026 (A)	2,100	2.700	2.500
Time of Test (Seconds)	198	मृह्म	720	234	204	1.5	1.8	3.6

(A) The normal rate used in tests in this report

(B) Minimum tensile strength, load indicating dial moved before recombing of data.

TABLE 23
VARIATION OF 1900" F TENELLE STRENOTE OF MASA ALLOY AND VARIOUS STRAIN RATES (NELT 418)

	S Elongation	00
Rolled & HT 64 Hrs. at 2200° F & 24 Hrs. at 1675° F	Ultimate Tensile Strength	62, 400 26, 400 29, 400
of a feet	Inches/Inch/Minute	.01 (A) .000. .000.

(A) This rate corresponds to normal rate used in all other tensile tests in this program.

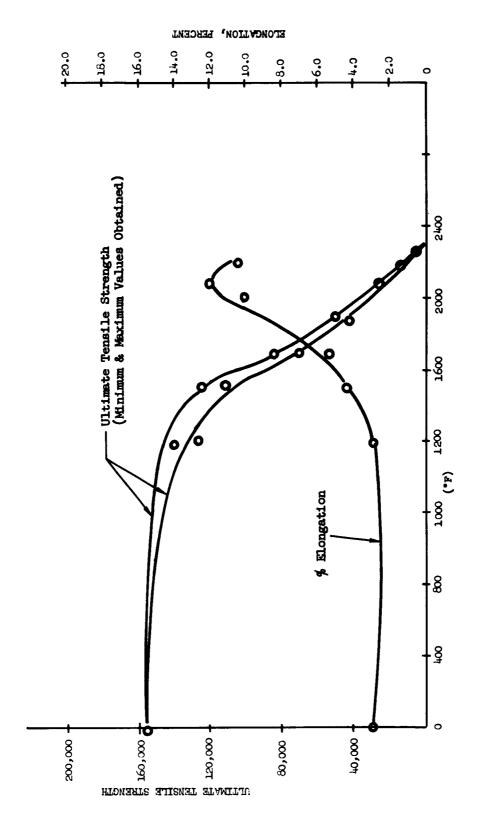


Figure 67. Ultimate Tensile Strength and Percent Elongation Variation of Inco 713c Rolled Sheet at Various Temperatures (Condition of Metal, Rolled + H.T. At 2150°F for 40 Hours + 24 Hours at 1600°F).

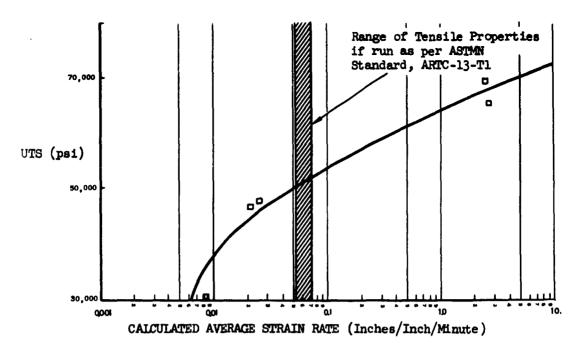


Figure 68. Calculated Average Strain Rate versus Ultimate Tensile Strength of Inco 713c Sheet at 1900°F. Condition, Rolled +HT 40 Hours at 2150°F + 24 Hours at 1600°F.

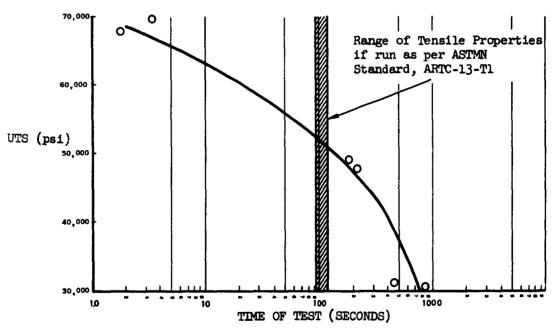


Figure 69. Total Time of Tensile Test versus Ultimate Tensile Strength of Inco 713c Sheet at 1900°F. (Conditions are the Same as Figure 68.)

2. Oxidation Resistance

a. Rate of Oxidation Attack

Oxidation tests in still air were run on NASA alloy, Inco 713c and No. 429 alloy in both as cast and rolled form. Oxidation tests were simultaneously run on Rene 41 sheet for comparison purposes. Table 24 and Figures 70 through 74 show the results of these oxidation tests. Briefly summarized, the tests show that Inco 713c alloy has about the same oxidation resistance at temperatures of up to 2100°F as Rene 41. NASA alloy has much poorer oxidation resistance than Inco 713c or Rene 41 and the No. 429 alloy is intermediate in oxidation resistance between NASA and Inco 713c alloys. Furthermore, the oxide scale formed on the NASA alloy has a marked tendency to explosively spall off of the metal surface upon cooling to room temperature, while the oxide scales formed on the other alloys are relatively adherent.

b. Identification of Oxidation Products

Samples were taken from the oxide scales formed on all of the alloys tested and these samples were analyzed by X-ray diffraction techniques. Table 25 gives the results of these analyses. It will be noted that the major constituent of the scale formed on the NASA alloy is NiO and the major constituent of the scale formed on the other two alloys is Cr₂O₃. This would indicate that chromium oxide is far more protective than nickel oxide. Since the Inco 713c has much better oxidation resistance than the No. 429 alloy, and since both oxide scales are primarily chromium oxide, it is obvious that other factors are also of importance in determining oxidation resistance.

3. Determination of Solidus Temperature

Nickel base superalloys have solidus temperatures much lower than their liquidus temperatures, and this wide mushy zone makes it difficult to determine solidus and liquidus temperatures by thermal analysis. In this program, an effort was made to determine the solidus temperature and to obtain an indication of the liquidus temperature by heating specimens of the alloy to temperatures within the temperature range expected to include solidus and liquidus temperatures and then water quenching these specimens. This was done with two alloys, NASA (Melt 387) and Ta-C (Melt 390)

TABLE 24 OXIDATION DATA

ge (mg/cm²) Alone 2100°F	-0.39- -0.37-b -1.37-b -1.19-b -0.13-b -0.88-b	-23.00- -21.40-b -8.50-b -28.53-b -7.14-b -7.14-b	+4.76- +5.34-b
Weight Change (mg/cm²) Specimen Alone 1900°F 2100°F	+0.17- +0.20-b	-24-8-4-8-4-5-4-5-4-5-4-5-4-5-4-5-4-5-4-5-	+6.68-• +6.32-b
Weight Change (ag/cm²) Specimen with Luose Oxide 1900'F	+0.51-8 +0.55-b +0.55-b +1.96-b +0.12-b +2.30-b	+6.76- +9.02-b +4.85-b +11.73-b +31.95-b +4.35-b +10.73-b	+4.54-e +5.33-b
Weight C Specimen w 1900 F	(a)-86-0+	-6.72- -	+6.37-b
TIME (min.)	3840 3840 180 180 100 100 1000 1000	3840 3840 180 180 100 100 100 100 100	3840 180 180
TEMPER- ATURE (°F)	1990 2000 2000 2000 2000 2100 2100	1900 1900 1900 21000 2100 2100 2100	1900 1900 2100 2100
CONDITION	as cast as cast as cast as cast as cast as cast as rolled 33% Reduction as rolled 33% Reduction as rolled 33% Reduction	as cast as cast as cast as cast as cast as cast as rolled 50%Reduction as rolled 50%Reduction 50%Reduction	as cast as cast as cast as cast
ALLOY	INCO 713C INCO 713C INCO 713C INCO 713C INCO 713C INCO 713C INCO 713C	NASA NASA NASA NASA NASA NASA NASA NASA	No. 429 Alloy

TABLE 24 (Continued)
OXIDATION DATA

2100 100 +7.48-b +2.78-b +2.78-b 2100 1000 +37.00-b 10.30.30
--

Footnotes:

(a) Specimen in open crucible.(b) Specimen in closed crucible.

TABLE 25

NO. 429, MASA ALLOY AND 713c X-RAY DIFFRACTION DATA RUN ON SCALED OXIDE

(Run on Both Powder Camera and Diffractometer)

Alloy and	Condition	Indicate	d Presence and M	ethod
		N10	c _{r2} 03	a-Al ₂ 0 ₃
No. 429	3 hrs. at 2100°F	Neither	Diffractometer indicates, major constituent	Diffractometer indicates presence
MASA	3 hrs. at 2100°F	Major constituent diffractometer & powder camera	Powder camera indicates possibilities of presence.	Neither method indicates presence
NASA	64 hrs. at 1900°F	Major constituent diffractometer & powder camera	Powder camera indicates possibilities of presence	Meither method indicates presence
713c	3 hrs. at 2100°F	Powder camera indicates possibility of presence	Major constituent powder camera only	
713c	64 hrs. at 1900°F	Powder camera indicates presence	Major constituent powder camera only	Powder camera indicates possibility of presence

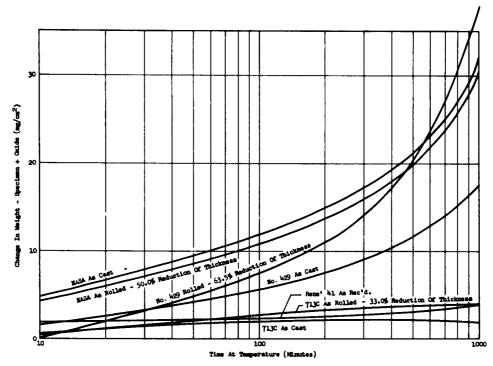


Figure 70. Increase in Weight of Specimen (With Oxide Scale) After Exposure at 2100°F.

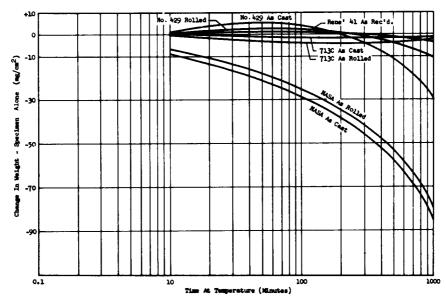


Figure 71. Net Change in Weight of Specimen After Exposure at 2100°F in Air. (After Removing Loose Oxide.)

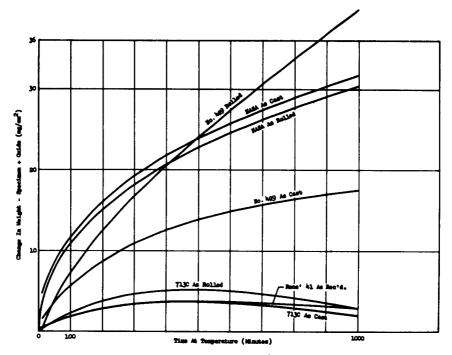


Figure 72. Increase in Weight of Specimen (With Oxide Scale) After Exposure at 2100°F.

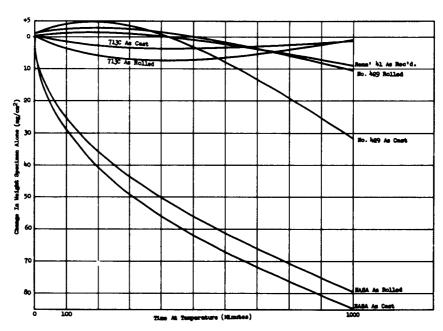


Figure 73. Net Change in Weight of Specimen After Exposure at 2100°F in Air. (After Removing Loose Oxide.)

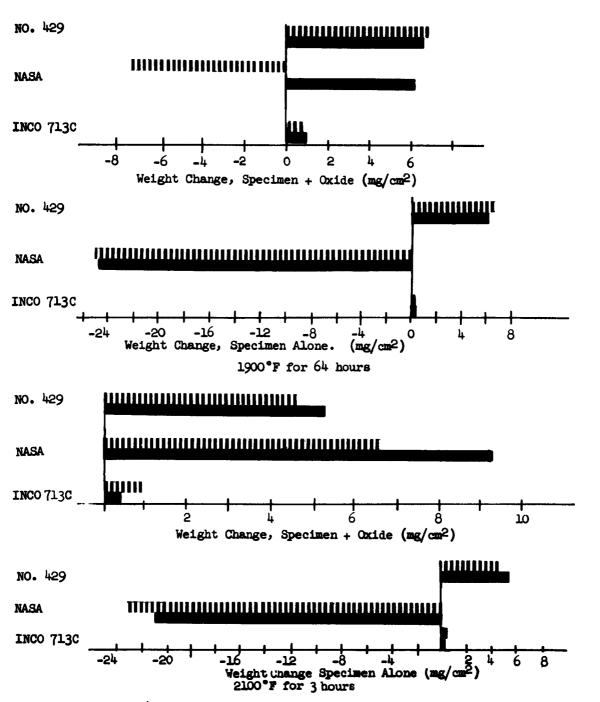


Figure 74. Oxidation Tests Run On As-Cast Inco 713c, NASA Alloy and No. 429.

modified Inco 713c. Figures 75 and 76 show the effects of heating these alloys to temperatures in the 2250°F and 2500°F range on the micro-structure of the alloys. It would appear from this data that the NASA alloy has a solidus temperature of between 2250°F and 2300°F and that the Ta-C modified Inco 713c has a solidus temperature of about 2250°F. Figure 77 shows photographs of the speciments heat treated in air in the 2250°F to 2500°F temperature range for these same two alloys. (It will be noted that the NASA alloy has a far greater tendency to scale at these temperatures than the Ta-C modified Inco 713c). It would also appear that the liquidus temperature of the Ta-C modified Inco 713c (Melt 390) approximates 2450°F and that the liquidus temperature of the NASA alloy is in excess of 2500°F.

4. Phase Identification

Both No. 429 and NASA alloy have large amounts of precipitated phase in their microstructure. Samples of both alloys were digested in chemical solutions designed to preferentially dissolve the matrix metal leaving the precipitate phase as an undissolved residue. Efforts to determine the chemical composition of the precipitate by X-ray diffraction means failed because the observed data did not match any of the compounds listed in the standard catalog. However, the specific gravity of the base alloys and of their precipitated phases were determined together with the percentages of precipitated phase. This data is summarized in Table 26.

5. Preliminary Fabricability Evaluation

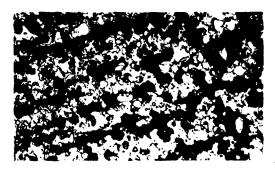
a. Investigation of Methods of Machining Test Bars

Initial efforts to machine tensile test bars from these nickel base alloys using high speed tool steel cutters were totally unsuccessful due to extremely rapid tool breakdown. Carbide cutters were successful in machining some of the nickel alloys, but resulted in a high proportion of ruined specimens in some other experimental alloys notably alloy No. 429. In all cases, tool wear was quite rapid and both tool and specimen vibration were serious problems. As a result of these difficulties, several other methods of tensile test specimen preparation were investigated.

A number of trials were made of electric spark discharge machining of alloy No. 429 using two different machines and a variety of settings on both machines.



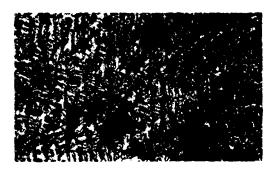
1/2 Hour at 2250°F, Melt 387, Water Quench.



1/2 Hour at 2300°F, Melt 387, Water Quench.



1/2 Hour at 2400°F, Melt 387, Water Quench.

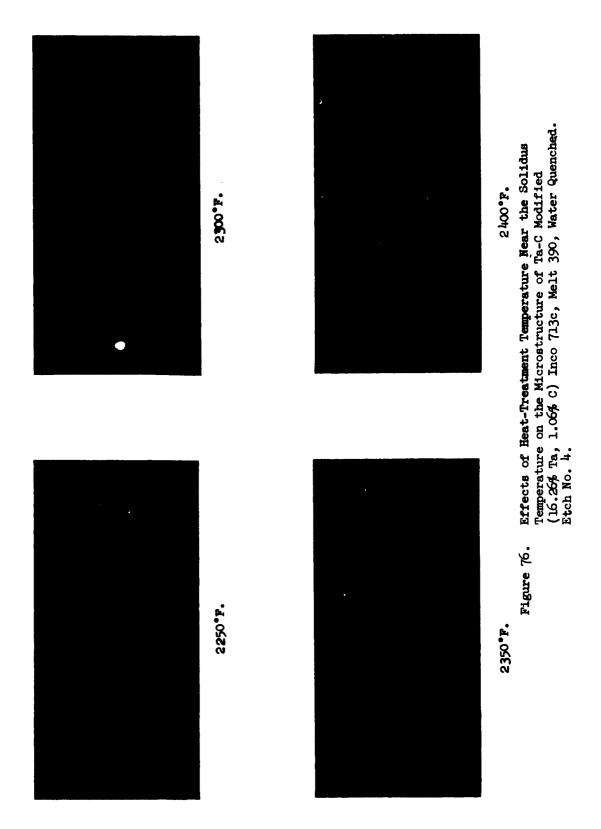


1/2 Hour at 2 450°F, Melt 387, Water Quench.



1/2 Hour at 2,500°F, Melt 387, Water Quench.

Figure 75. Effects of Heat-Treatment Temperatures Near the Solidus Temperature on the Microstructure of NASA Alloy. Etch No. 4. 100X.



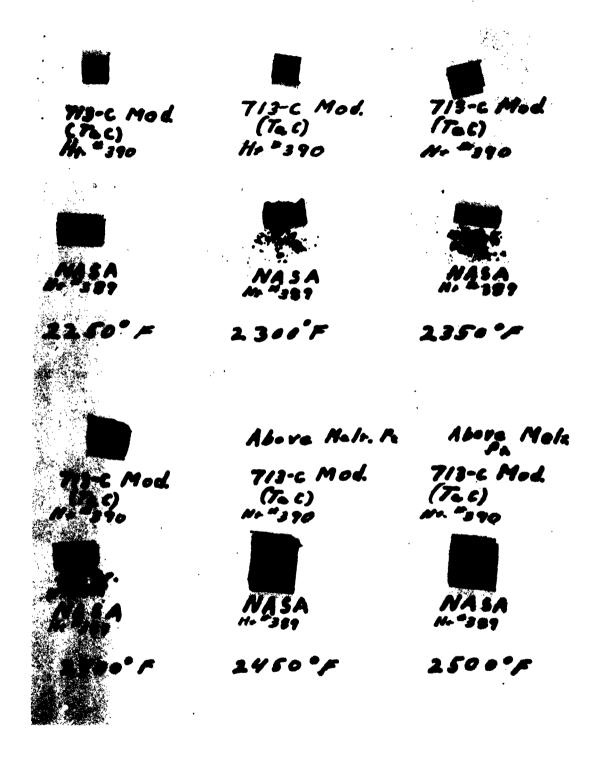


Figure 77. Specimens Heated to Various Temperatures for One-Half Hour and Quenched to Determine Solidus Point for Two Alloys.

TABLE 26
SPECIFIC GRAVITY OF NO. 429 AND MASA ALLOY

Alloy and Condition	Specific Gravity	Remarks
No. 429 as cast	8.847	
No. 429 precipitated phase a	7.2 8 4	35% by wt. 42.4% by volume
No. 429 dissolved portion ^b	9.97	65% by wt. 57.6% by volume
MASA alloy as cast	8.397	
MASA alloy rolled	8.472	_
WASA alloy precipitated phase ^a	4.041	13.9% by wt. 28.8% by wolume
MASA alloy dissolved portion ^b	10.12	86.1% by wt. 71.2% by volume

a The residue collected on 2 micron filter after dissolving as cast specimens in concentrated HCl+ferric chloride solution

b By calculation

A variety of cutting electrode compositions were tried including the following:

- 1. Brass
- 2. 4340 Steel
- 3. Graphite
- 4. Silver-tungsten carbide alloy

All of these electrodes eroded rapidly during the test without substantially cutting the No. 429 alloy. This investigation failed to develop any practically useful methods of electric spark discharge machining of this alloy.

A number of test specimens were successfully produced by the following grinding procedures:

- Saw blanks out with a reinforced alundum cutoff wheel (Norton No. TBNX24) under a flood of coolant.
- Rough grind to test bar contour by hand on a silicon carbide grit bench grinder wheel.
- Belt grind to approximate final contour using a silicon carbide grit belt.
- 4. Finish grind to size on a surface grinder using a silicon carbide grinding wheel.
- Drill required holes with a carbide tipped drill.

The most rapid procedure developed to date for cutting tensile test bars from these nickel base alloys is as follows:

- Saw blanks with a silicon carbide cutoff wheel under a flood of coolant.
- 2. Using a belt grinder of special design constructed for the purpose at Vought, Figure 78, cut the contour of the test bar in a single operation using wet or dry, 40 grit, silicon carbide, abrasive belts under a flood of coolant.
- Drill required holes with a carbide tipped drill using a rigid drill fixture to position the drill.



V. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

The work done under this contract has resulted in the following developments:

- 1. It has been demonstrated that thin cast slabs of nickel base superalloys can be directly hot rolled into sheet without the need for intermediate forging or extrusion operations. This greatly simplifies the procedure for obtaining sheet of a new cast alloy.
- 2. Inco 713c rolled and heat treated sheet has a 1900°F ultimate tensile strength of about 50,000 psi at strain rates less than ASTM standards, with good room temperature strength and ductility, and good oxidation resistance. This represents about a 200°F increase in useful temperature over the best previously available superalloy sheet, Rene' 41. The rate of strain during tensile tests has a substantial effect on the tensile strength reported, especially 1900°F. The strain rate in the elastic range used in most of the tensile tests in this program was calculated to be less than .005 in/in/minute as established by ASTM standard procedure. (Note: See Appendix C, pg.123). Higher strain rates were observed to give higher strengths.
- 3. NASA TaZ8 rolled and heat treated sheet has a 1900°F tensile strength of about 50,000 psi with poorer ductility and oxidation resistance than the Inco 713c sheet. On the other hand, NASA TaZ8 alloy has a higher recrystallization temperature and hence might have higher stress rupture strength at temperatures of 1900°F and above.
- 4. Nickel base alloys can be strengthened by precipitation hardening using complex refractory metal carbides as the precipitating constituent rather than nickel aluminide.
- 5. The use of a rigid rolling mill, which does not depend on hold down screws for positioning of the movable roll, makes it possible to roll metals and alloys having lower ductility at rolling temperature than is possible with rolling mills of conventional design. Indications are that this mill is capable of rolling high temperature alloys down to foil thickness.
- 6. Long time solution heat treatments gave the best improvement in the ductility and tensile strength of Inco 713c of all the heat treatments tried. The strength and ductility of

- of NASA alloy is affected more by aging heat treatments than solution heat treatment.
- 7. A limiting factor in this program, with respect to further improvements in alloy compositions, was the crucible materials employed. Specifically, when high percentages of tantalum were added to melts, reaction between the molten metal and the crucible occurred.
- 8. Thin cast slabs of Inco 713c can be directly rolled into 25 mil sheets up to 10 x 16 inches in size.

B. Recommendations

- 1. It is recommended that the basic techniques of casting thin slabs into sheet be applied to the new NASA nickel base alloys and to the refractory metals. It is believed that this technique will permit a great reduction in the time required to get a new alloy from discovery to commercial use.
- 2. The tensile properties of Inco 713c sheet obtained in this program indicate that its strength at 1900°F offers the potential of a substantial advancement over presently available nickel base alloy sheet and foil. It is recommended that investigations be initiated to obtain additional mechanical, and physical property data needed for further evaluation purposes. These investigations should include tensile and stress rupture strength, notch sensitivity, tensile strength as a function of strain rate, compressive strength, and limited fabricability evaluations.
- 3. The complex refractory metal carbide precipitation hardening system used in alloy No. 429 should be further investigated as a possible means of producing superalloy sheet which would have usable strength at temperatures up to 2400°F combined with good oxidation resistance.
- 4. Improved melting furnace equipment is urgently needed to eliminate crucible-metal reactions. One possible approach is the use of water cooled metal crucibles for melting the alloy prior to casting.

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APPENDIX A

MELTING AND CASTING TECHNIQUES AND EQUIPMENT

A. Melting Techniques

All melting of nickel alloy done in this program was done in the Vought designed Plasma-Resistance furnace. The particular furnace used in this program is shown in Fig. 79 and has a melting capacity of about 25 pounds of nickel base alloy. This furnace operates at all times under an argon atmosphere which can be at atmospheric pressure or at reduced pressures down to 30 microns. All melting operations done under this contract were performed under an atmospheric pressure of argon, although the argon pressure in the furnace was reduced to the 30 to 1000 micron level for the casting operation. In addition to melting under an argon atmosphere, argon gas is bubbled through the melt from the time melting commences until pouring operations start. The purpose of this is twofold: one. to mix the alloying constituents in the melt; and two, to remove gaseous impurities, such as oxygen, hydrogen, and nitrogen. (See Table 10, page 37).

The heat source for preheating the furnace prior to charging of the metal is a low voltage, high amperage carbon arc. The arc length under these conditions is approximately 1/2 inch and in general the arc behaves normally in spite of the argon atmosphere. After the furnace interior reaches a temperature of 2800°F, the electrical characteristics of the argon gas change. As a result of this change, the gap between the electrodes is increased to about 8 inches without increasing voltage or decreasing amperage. The consumption of the graphite electrodes also essentially ceases. Under these conditions the flow of current through the furnace can be interrupted and restarted without change in the electrode position, and without the use of high frequency or high voltage starting currents. Experience with this furnace over a period of time has shown that alloys melted under these conditions will not pick up any carbon from the furnace atmosphere. In fact, carbon losses are frequently encountered due to the removal of oxygen from the melt, apparently as carbon monoxide.

During this program both magnesia and zirconia refractory crucibles have been used in this furnace for melting nickel base alloys. In both cases, there is evidence that there is some reaction between the molten metal and the crucible. In the case of the zirconia crucible, this reaction results in an increase in the zirconium content of the metal. Since zirconium is generally considered to be a desirable alloying element in nickel

base alloys in small percentages, this reaction is not detrimental unless excessive amounts of zirconium are added. In the case of the magnesia crucible, magnesium metal and boron are picked up by the metal as a result of the crucible reaction. Magnesium metal has a high vapor pressure at the temperatures used in melting nickel alloys, and for the most part is vaporized. The small amount of residual magnesium left in the alloy (See Table 7) does not appear to be seriously detrimental. The boron pickup in the alloy is in the range generally considered to be desirable for nickel base superalloys. (See Table 8). Therefore, for the particular nickel base alloys primarily concerned in this program, both magnesia and zirconia crucibles appear satisfactory. (Tables 7 and 8 on pages 36 and 37).

The crucibles used in this program were all fabricated at Chance Vought from purchased refractory grain. The refractory materials used in this program are identified in Table 9, page 37. The crucibles were fabricated by the following process:

- a. Mix dry grain with water to form a damp sand.
- b. Hard ram or press the damp refractory into a steel flask around a wood form to form the crucible shape desired.
- c. Place the rammed crucible on a flat silicon carbide plate and remove both the wood form and the steel flask.
- d. Dry the crucible on the silicon carbide plate for four hours at 500°F.
- e. Fire the dried crucible in an air atmosphere in a furnace whose temperature is gradually raised from 500°F to 2500°F over a period of 8 hours.
- f. Furnace cool the crucible and install in the melting furnace.

Of these crucibles, those made from Magnorite X were the most generally satisfactory and had the longest service life. The zirconia crucibles were relatively fragile and subject to damage from thermal shock. As a result, their life in the furnace was quite limited. The crucibles made from RM 1152 tended to soften at service temperatures and underwent post-firing shrinkage. This caused early failure of the crucible roof with resultant short crucible life.

In most of the alloys melted, crucible reactions were not serious problems. However, in the few high tantalum nickel base alloys melted crucible reactions were very serious and resulted in the removal of much of the tantalum from the alloy and serious damage to the crucible. (See Table 6, page 36). Melt Nos. 395 and 396 are not shown in Table 6 because they resulted in the loss of both crucible and

melt and hence no analysis of the melts was possible. Melt Nos. 395 and 396 had 40.56% tantalum and a total of 43.08% refractory metal content. The trend in improving superalloys appears to be in the direction of increasing refractory metal content. It is apparent that any substantial increase in refractory metal content is going to require the development of improved crucible materials or different melting techniques. Water cooled metal crucibles may be an answer to this problem, although conventional consumable arc melting of ingots in water cooled copper crucibles does not appear to be a satisfactory answer.

The general procedure used in making all melts in this program is as follows:

- a. Preheat the melting furnace to about 3000°F.
- b. Charge and melt the major constituent. In alloys being made from individual constituents, this would be nickel. In alloys made by modifying an existing alloy, this would be the starting alloy; for example, purchased Inco 713c yacumm melted bar.
- c. Charge and melt alloying constituents, adding the refractory metals first and the volatile elements last.
- d. After all alloying additions are made, maintain the melt at temperature and bubble with argon gas for 15 to 20 minutes to thoroughly mix the alloy and to remove gaseous impurities.
- e. The metal is then raised to pouring temperature and poured.

B. Casting Technique

All castings made in this program were made in the plasma-resistance melting furnace using preheated ceramic molds press formed on a molding press. This molding and casting technique permits the casting of relatively large, thin sheets of nickel base superalloys, as well as a wide range of other alloys.

The ceramic molds used in this program were press formed on a molding press from both silica and zircon base refractory powders. No evidence of metal-mold reaction has been found with either molding composition, if pouring temperature is controlled in the 2700-2900°F range. In practice these powders are mixed with ceramic and chemical bonding agents in minor percentages, dampened to form a workable material, and pressed into mold halves under a mold face pressure of about 500 pounds per square inch. The pressed mold half is quite weak at this point and must be cured prior to further handling. This curing is normally done by baking the mold at from 300 to 500°F for

3 to 8 hours depending on size and the particular bonding materials being used. This cured mold is quite hard and strong and can be sawed, drilled, or otherwise machined as may prove advisable. Through this program the gates and risers were machined into the cured mold. Initially the cavity for the formation of the sheet casting itself was formed into the mold at the time of pressing by use of a pattern. Fig. 83 shows such a pressed and cured mold half. Later in the program castings were made in molds where the cavity for the sheet itself was also machined. The cured ceramic molds are easily and rapidly machined using carbide tools or abrasive wheels and belts. It was found that under some conditions some slight warpage and distortion of the molds occurred during curing. It was found that the highest degree of uniformity of thickness in the cast sheet could be obtained by machining the sheet cavity after curing of the mold. No further warpage of the mold during the preheating and pouring operations has been observed.

The cured and machined mold halves are assembled into stacks suitable for pouring, and the outside surfaces of these mold stacks are coated with a slurry of refractory grain, southern bentonite, and water. The purpose of this coating is to prevent the mold helves from shifting relative to each other during handling, prehest and pouring. These mold stacks are placed in the preheat furnace and preheated to from 1200°F to 1800°F on a pre-determined heating cycle. This heating cycle is calculated to permit the uniform heating of the mold stack without mold cracking due to thermal shock. (See Fig. 84). The preheated mold stack is placed in the melting furnace, which contains a charge of molten metal ready for pouring and clamped in place. Fig. 80 shows the mold stack in this position. The vacuum tight access door is closed on the furnace and the pressure inside the furnace chamber reduced to from 30 to 1000 microns. Fig. 81 shows the furnace in this condition. The entire furnace chamber is then rapidly rotated 180 degrees, almost instantly pouring the molten metal charge through a 3 inch diameter pouring neck into the evacuated mold. Atmospheric pressure of argon gas is then restored in the furnace chamber to accelerate mold cooling. This combination of almost instantaneous pouring with the use of a hot evacuated mold permits the filling of relatively large areas of sheet down to 30 mil thicknesses. Fig. 82 shows the furnace in the poured position. After pouring the furnace remains in the poured position for about 15 minutes to be certain that the casting has completely solidified. At the end of this time the furnace is returned to its original position, the vacuum tight access door is opened, and the poured mold stack is removed.

The poured mold stack is allowed to cool in air to room temperature. It has been found that this minimizes warpage and distortion of the cast sheet. After cooling to room temperature the ceramic mold is broken away from the casting and the cast sheets are sawed off from the gates and risers. These cast sheets are sand blasted, X-rayed, Zyglo inspected, and are then ready for rolling. The "tree" of

cast sheets after the ceramic mold is broken away and before the sheets are sawed off, is shown in Fig. 85. Fig. 86 shows another larger cast sheet with the riser still attached. This sheet is about 0.1 inch thick and approximately 8" x 16" in size. This larger sheet represents the largest mold which can be made on the existing molding press at Vought but is not believed to represent any maximum size limitation of the process as a whole. The risering shown in Fig. 86 gives a higher degree of X-ray soundness in the sheet as a whole than does the risering shown in Fig. 85. Fig. 86 represents a later risering procedure.

Efforts have been made to roll thicker cast slabs and slices from ingots in this program. These efforts have been unsuccessful, demonstrating the practical need for casting the starting sheet in thicknesses approximating 0.1 inch.

This necessity of starting with thin cast sheets has a good technical explanation. Figure 87 shows a typical simple eutectic type of phase diagram. The vertical dashed line represents a particular alloy composition being cooled from the liquid state to room temperature. As this alloy cools in the form of a sheet casting (Shown in Fig. 88) the first crystallites of solid metal to form are composition A. The remaining liquid at this point has a composition of A'. If cooling continued slowly enough for equilibrium conditions to occur, the composition of the solidified metal would be consecutively represented by B, and C plus C'. Under conditions of rapid solidification, as it occurs in a casting, the first metal to freeze has composition A. As cooling continues under these conditions, the composition of subsequent layers of metal deposited on the initial crystal of metal have compositions represented by B and C and the points of the solidus curve intermediate between these points. As this metal freezes out of the liquid under these non-equilibrium conditions the composition of remaining liquid shifts to the right of the equilibrium compositions represented by B' and C'. The last metal to freeze will be in the grain boundaries, and will have a composition represented by a point to the right of C'. Figure 89 shows a simple solid solution phase diagram with a dashed line representing a particular alloy composition cooling from above the liquidus temperature to room temperature. The actual effect on the metallurgical structures of the casting remains the same as is shown in Figure 88. The significance of all this is that any casting of an alloy composition will have substantial variations in chemical composition within each grain, and that the grain boundaries always represent the minimum melting point composition. The diagrams shown are for relatively simple phase diagrams. In the case of more complex phase diagrams, particularly those involving peritectic reactions, the depression of the melting point at the grain boundaries can be even greater. Diffusion is extremely rapid in liquid metals and relatively speaking, very slow in solid metals at any temperature. Therefore, the larger the grain size of the casting the greater the degree of macrosegregation

resulting from the mechanism of solidification. The larger the section cast, the slower will solidification take place, and the larger the grain size. With this larger grain size a larger zone of low melting point constituent will be formed at the grain boundaries. In nickel base alloys at least, this lower melting point grain boundary constituent appears to be substantially weaker at elevated temperatures. Hence, when efforts are made to hot roll these alloys cast in relatively thick sections, failure occurs along the grain boundaries.

In any alloy casting there will be some degree of segregation. If the casting is relatively fine grained and is a nickel alloy, this cast alloy sheet can be hot rolled. Figure 90 shows the relative structure of the cast alloy as shown in Figure 88 after it has received a substantial amount of reduction in thickness by rolling. The S spacing between the centers of segregation has been greatly reduced from Figure 88 to Figure 90 by virtue of the rolling operation even without any effect of heat treatment. Figure 91 and 92 show these same alloys after heat treatment. It will be noted that the physical form of the segregation has tended to change and to be diminished. However, even after heat treatment the rolled structure has a closer spacing, S, between points of segregation. Also the rolled structure has the higher melting point constituents lined up in the direction of rolling, while in the cast structure the higher melting point constituents are lined up normal to the sheet surface. As can be seen in Figure 93 alignment of segregation in the cast structure favors early failure under tensile stress and the path of crack formation is short. This tends to result in low tensile strength and ductility, particularly at room temperature. Figures 94 and 95 show a rolled structure under tensile load. The strong segregated structure in this case is in the direction of stress and the path of crack formation is relatively long. This favors higher tensile strength and greater ductility. Furthermore, in the heat treated rolled structure, since the S spacing is already much smaller as a result of rolling, the time to effect equilibrium conditions by diffusion is far shorter than it would be on an as cast structure. Since the rate of diffusion for substitutional elements is usually an exponential related to distance, this smaller S value for the rolled alloy can result in an adequate heat treatment time for a rolled alloy being serveral orders of magnitude less than it is for a cast alloy. In both cases, there is no effective substitute for keeping original macrosegregation to a minimum by rapidly freezing the metal in a fine grain size. In the case of complex nickel base alloys, this would appear to be best done by casting thin sheets.

The proper selection of mold preheat and metal pouring temperatures are vital to the achievement of useful castings. In the case of the NASA TaZ8 alloy, it was found that a mold preheat temperature of 1800°F resulted in serious hot tearing of the sheet casting while mold preheat temperatures of 1600°F and below resulted in cast sheets

free of hot tearing. Excessively high or uncontrolled pouring temperatures resulted in mold-metal reactions that damaged both the surface finish and the properties of the cast sheet. In general, the optimum conditions for casting sheet from nickel base alloys has been found to be a mold preheat temperature of from 1200 to 1600°F and a metal pouring temperature of about 2800 to 2850°F.

Most castings made in this program were made in silica base molds because of previous good experience in casting nickel base alloys in molds of this composition. Silica base molds have a greater tendency to crack due to thermal shock during mold preheat than zircon or alumina base molds. Therefore both alumina and zircon base molds were used for a limited number of melts in this program. As a part of this investigation a series of Inco 713c melts were cast into zircon molds. All of these castings showed substantial gas porosity. This is thought to be due to occluded gas in the mold caused in part by the very fine particle size of the available zircon. The original castings had been made in silica molds and when the use of silica molds was resumed with melt No. 544 the gas porosity problem disappeared. Earlier zircon molds made with coarser zircon flour had been successfully used to make NASA alloy castings. This tends to corroborate the gas occulusion explanation for the gas porosity in the Inco 713c castings made in fine zircon flour molds.

Figure 79. Melting and Casting Furnace in Empty Position.



- 1. Furnace shell containing the crucible.
 2. Vacuum tight door.
 3. Chamber for containing the mold stack during pouring.
 4. Air clamps for clamping the mold in place during pouring.
 5. Vacuum hose connection to vacuum pump for evacuating the furnace chamber.
 6. Fixed support stand in which the furnace shell rotates during pouring.
 7. Machined face of the furnace against which the vacuum tight door (2) fits during the pouring operation.
 8. Electrode holder electrically insulated from the main shell.
 9. Trunnions of the furnace which rotate during the pouring operation.

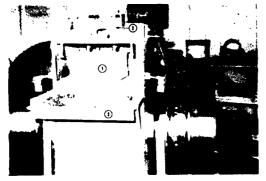


Figure 80. Melting and Casting Furnace with Mold in Prepouring Position.

- Hot mold stack clamped in pouring position.
 Air clamps clamping the mold in position.
 Vacuum tight door.

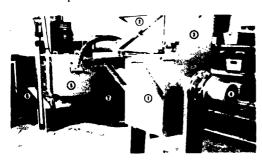


Figure 81. Melting and Casting Furnace Loaded and Ready to Pour.

- 1. Furnace shell containing the crucible.
 2. Vacuum tight door locked in place and furnace chamber evacuated.
 3. Portion of the furnace shell containing the hot mold stack.
 4. Vacuum bell seals over the electrode holders.
 5. Old trap in the vacuum line.
 6. Mechanical vacuum pump used to rapidly evacuate the furnace chamber.
 7. Welding transformer used to supply power for the melting furnace.





Figure 82. Melting and Casting Furnace in the Poured Position.

- Portion of the furnace shell containing the hot poured mold stack.
 Vacuum bell seals over the electrode holders.
 Argon inlet line into the furnace chamber.
 Vacuum hose connection to the furnace chamber.
 Furnace shell containing the crucible.

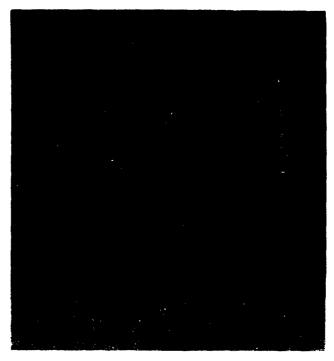


Figure 83. Pressed and Cured Ceramic Mold Half.

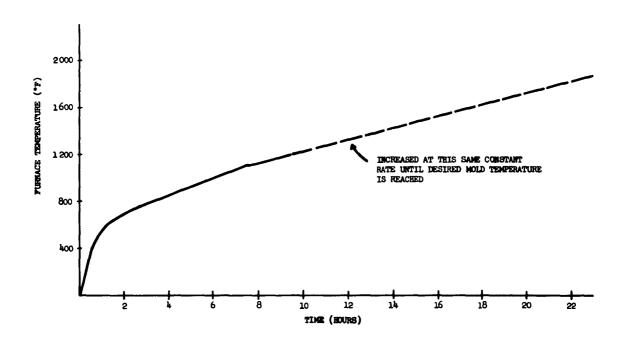


Figure 84. Typical Mold Preheat Cycle.



Figure 85. Cleaned Casting with Cast Sheets Still Attached to the Riser.

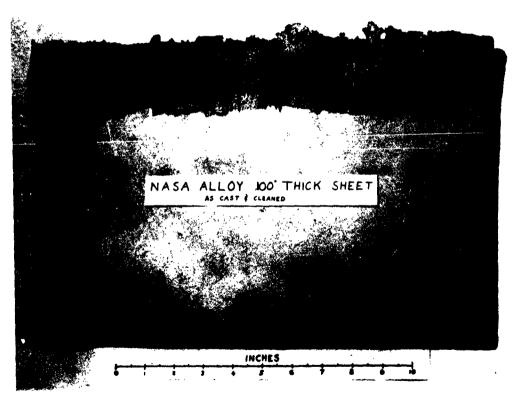


Figure 86. NASA Alloy .100" Thick Sheet (as Cast and Cleaned).

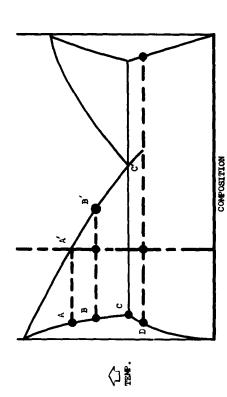


Figure 87. Solidification in a Typical Simple Eutectic System.

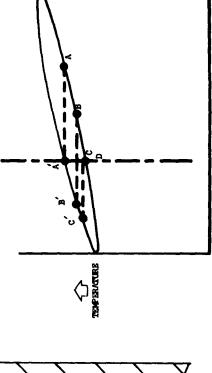
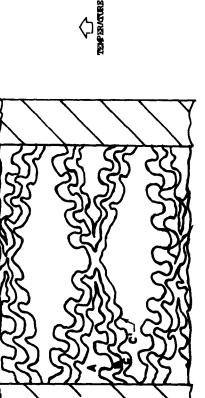
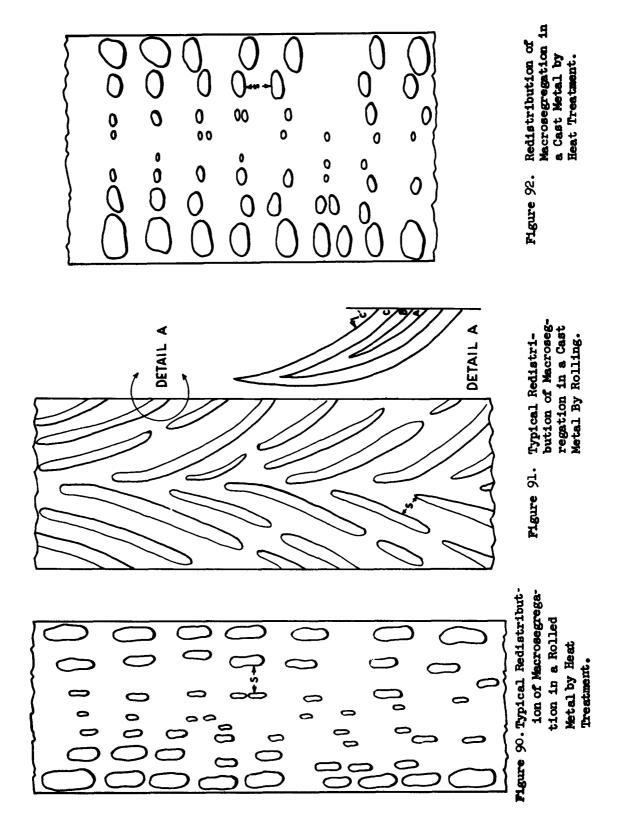


Figure 86. Typical Solidification Mode in a Casting.



in a Figure 89. Solidification in a Simple Solid Solid System.







Tensile Failure in a Rolled and Heat-Treated Structure. Figure 95.

APPENDIX B

THE RIGID ROLLING MILL

A. Statement of the Problem

Difficulties encountered in rolling nickel alloy sheet on conventional rolling mills caused Voughtto design and construct a rolling mill of new and unique rigid design in support of this program. Many of the problems encountered in rolling of nickel base alloys on conventional mills appeared to be related to "crowning" of the sheet during rolling and to an inability to adequately predetermine and control the position of the movable roll during the rolling operation. Crowning of the sheet occurred in the early stages of rolling nickel alloy sheet on conventional mills. In subsequent passes the thicker section in the center of the sheet tended to elongate more rapidly than the outside thinner sections. This put the outside sections under tensile stress and this in turn caused serious edge cracking which soon penetrated to the center of the sheet. This problem could be minimized by a process of repeatedly removing the thinner outside edge sections of the rolled sheet by sawing them off. However, this rapidly becomes self-defeating as the width of sheet available for rolling diminishes to the vanishing point. The inability to control the position of the movable roll contributes largely to the crowning problem and also makes it impossible to provide a uniform reduction per pass over the entire sheet of material being rolled. As a result there are substantial variations in the thickness of the rolled sheet even aside from the crowning problem. These variations contribute to later cracking problems. An analysis of the problem of rolling mill design shows that "crowning" of rolled sheet can be caused by two different conditions as follows:

- 1. Bending of the rolls caused by the separating force of the metal sheet passing between the rolls. For 6" diameter steel rolls, rolling nickel base superalloy sheet 12 inches wide, this crowning can be calculated to be approximately 3 mils maximum. The experimental results obtained on the rigid mill at Vought corroborate this quite closely.
- 2. Random, uncontrolled motion or cocking of the movable roll due to the inability of the hold down screw to seat precisely the same way each time or due to unequal deflection of the hold down screws. This feature of conventional mills has been observed to cause crowning of up to 20 mils in a 4 inch wide sheet.

The Vought mill was therefore designed to eliminate the hold down screw method of construction with all of its resulting problems.

B. Description of the Rigid Mill

The rigid mill can be used either as a two high mill with 6 inch diameter working rolls and no back up rolls, or as a four high mill with 1 1/2 inch diameter working rolls and 6 inch diameter back up rolls. This rolling mill is driven by a 30 horsepower electric motor operating through a chain drive. All four rolls in the four high configuration are driven. Figs. 96 and 97 show the rigid rolling mill and associated metal preheat furnace. The significant characteristics of the construction of this rolling mill are summarized below:

- a. The mill can be used as either a 2 high or a 4 high mill.
- b. In the 2 high configuration, the working rolls are 6" in diameter with a 12" working length. The rolls are hardened steel and the bearings are bronze sleeves having 4" ID.
- c. In the 4 high configuration, the working rolls are 1 1/2 inch diameter hardened steel with brass bushing blocks for bearings. The back up rolls are the same 6 inch diameter rolls used as working rolls in the 2 high configuration.
- d. The mill is capable of withstanding a separating force of approximately 900,000 pounds. The separating force experienced is recorded by means of suitable instrumentation connected to strain gauges attached to the columns of the mill. The maximum separating force experienced to date has been 167,000 pounds.
- e. The column area is 29.6 inches. The rolling mill does not have any hold down screws so this column area directly reflects the true rigidity of the mill.
- f. The maximum mill deflection at 900,000 pounds separating force is approximately 40.5 mils if no preloading is applied. The maximum mill deflection at the maximum separating force incurred to date (167,000 pounds) is approximately 7.5 mils if no preloading is applied. The maximum preloading force used to date has been 51,900 pounds. With this preload, a separating force of 62,280 pounds experienced in rolling 3 mil foil 3 inches wide corresponds to a mill deflection of .05 mils. The working rolls in the present rigid mill are 4340 steel hardened to Rc 38. When rolling superalloy foil, these rolls plastically deform, and this plastic deformation is the present limiting factor in reducing foil gauge. Tool steel or carbide work rolls would be expected to make possible the rolling

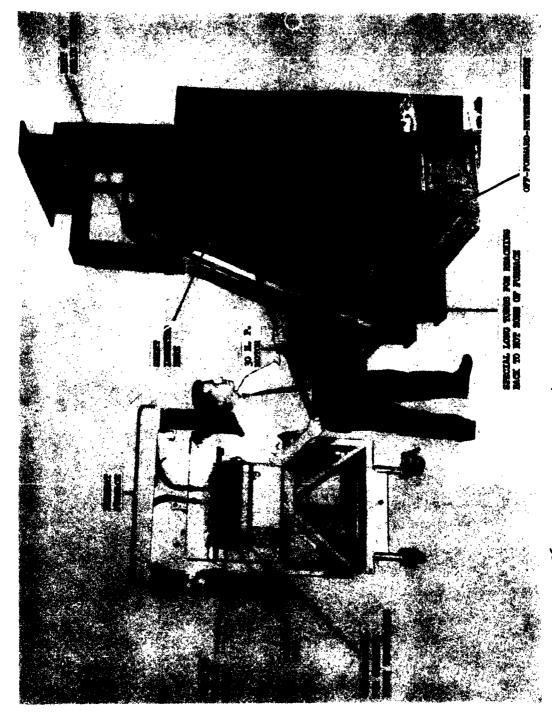


Figure 96. View of Vought Rolling, Mill and Preheat Furnace.

Figure 97. Vought Rolling Mill in 4-High Configuration.

of thinner gauge foil.

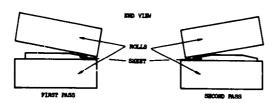
- g. Roll speeds of 97.5, 139.3 and 166.0 surface feet per minute are currently usable on the rigid mill. Other roll speeds can be obtained by changing one of the main drive sprockets, if desired.
- i. The position of the movable roll is determined by placing solid shims of predetermined thickness between the main bearing blocks of the movable roll and the mill frame.
- j. Preloading of the mill is accomplished by the use of tapered wedge blocks driving the movable roll toward the fixed roll.

C. Discussion of the Advantages of the Rigid Mill

This mill differs substantially from conventional rolling mills in several important ways. Rolling mills consist of two rolls or sets of rolls; one of which can be translated relative to the axis of rotation of the other roll or set of rolls. Both sets of rolls are rotated toward each other at the same speed. In a conventional rolling mill the movable roll or set of rolls is moved and then held in place by means of hold down screws connecting the bearing blocks of the movable roll with the rolling mill frame. In order for a screw to move it must have some clearance between the male and female portions of the screw mechanism. Due to this clearance, a screw will seat differently under load each time it is loaded, particularly if the load is not truly axial at all times. In a conventional rolling mill no load is normally applied to the hold down screw until the metal enters the rolls. Depending on how the metal enters the rolls, how uniform the entering metal is in thickness, the degree of metallurgical uniformity throughout the metal, and a number of other possible variables, the rolling load of the mill will be transmitted to the hold down screws in varying degrees of inequality and non-axiality. As a result, the true position of the movable roll with respect to the fixed roll can vary appreciably depending on how each of the two hold down screws may happen to seat under the load as applied. Hence, the movable roll may cock in one direction during one pass and in the opposite direction during the next pass. (See Fig. 98)

The hold down screws normally have a relatively small cross sectional area relative to the loads being applied and relative to the size of the mill frame. Hence, these hold down screws will compress elastically under load. Since this compression is elastic in nature, it will be proportionate to the particular load being momentarily applied to the particular hold down screw in question. Since the hold down screws are of relatively light construction, the elastic strain incurred will be a substantial portion of the total strain incurred in the mill as a whole. Both the random seating of the

hold down screw and its elastic deformation in compression serve to apply maximum rolling load to first one side of the sheet and then in subsequent passes to the other side of the sheet. This is the primary cause of "crowning."



SHEET NOLLS

Figure 98. Diagram Showing Crowning as a Result of Two Successive Passes on Conventional Mill.

Figure 99. Diagram Showing Crowning Caused by Bending of the Rolls.

A secondary, but very minor, cause of crowning is the actual deflection of the roll itself. (See Fig. 99) When the actual rolling loads are determined experimentally by strain gauge measurements and the true deflection of the roll by bending during rolling is calculated, it is found that, for a 6 inch diameter working roll with no back up and with a 12 inch working width, bending deflection accounts for only a maximum crown of about 3 mils. This corresponds to the actual crown measured on an 11 inch wide sheet of Inco 713c rolled on the rigid mill. On a conventional mill 10 to 20 mils of crown was observed on a 4 inch wide sheet.

APPENDIX C

ELEVATED TEMPERATURE TENSILE TEST PROCEDURE

The test procedure used in tensile testing nickel base alloy sheet at elevated temperature is described and discussed below:

- A. A sheet tensile test specimen is gripped in the tensile test machine and three 36 gauge chromel-alumel thermocouples are spot welded to the surface of the specimen, one thermocouple being spot welded at each end of the gauge section and one at the center of the specimen. Each thermocouple consists of one chromel and one alumel wire, each separately spot welded to the specimen in close parallel position having the same longitudinal position on the specimen. The purpose of this is to assure the measurement of the specimen temperature and not just the temperature of a projecting bead. The thermocouples are connected to a Brown multipoint high speed strip chart recorder. Heating conditions are adjusted until all three thermocouples read the same temperature. Once test conditions have been standardized for a given set of specimens, further tests are conducted using only the center thermocouple in accordance with Standard Test Procedure ARTC-13-T-1, Rev. 6/1/59.(6)
- B. In gripping thin sheet metal specimens great care must be taken to assure that the method of gripping does not adversely affect the axiality of loading. When pin type grips are used, the specimen should be finally fastened in place while under a small tensile load with the specimen at room temperature. Otherwise, it is possible to cock the specimen in the grips during tightening of the pin bolt, resulting in nonaxial loading of the specimen.
- C. Where only tensile strength and elongation are required, elevated temperature tensile tests are run without the use of an extensometer. Hence, it is impossible to directly determine strain rates. Test machine head travel rates are not an adequate indication of strain, particularly during the early stages of the test. The reason for this is that the gripping and linkage devices used to attach the test specimen to the test machine strain when the testing load is applied to the specimen, and these may have a total strain substantially greater than that of the gauge length of the specimen.

In these tests, a constant loading rate of 16,000 pounds per square inch of test specimen per minute was used. Data published by Haynes Stellite for Rene' 41 (8), a nickel base superalloy, gives values of elastic modulus for this alloy of 16,200,000 at 1800°F and 8,200,000 at 2000°F. It would appear reasonable to assume an elastic modulus of about 12,000,000 at 1900°F. A load rate of 16,000 psi on a nickel alloy having an elastic modulus of 12,000,000 corresponds to a strain rate in the elastic range of 0.0013 inches per inch per minute. The standard strain rate specified for use with an extensometer is .005 inches per inch per minute. Therefore, the strain rate in the Vought tests would result in slightly lower reported strength values than the standard test using an extensometer. The Vought test results in a testing time of from 3 to 4 minutes. The procedure described in ARTC-13-T1 results in a testing time of from 1 1/2 to 2 minutes. ASTM testing procedure results in a testing time of from 1.2 to 2.1 minutes. The Vought testing procedure would, therefore, be expected to give slightly lower test results than the standard procedures cited.

D. After the specimen is broken, total elongation is determined over a one inch gauge length by a caliper measurement.

APPENDIX D COMPLETE CASTING DATA SUMMARY

	Remarks												નું	÷	÷	
	Hold Temp.	1400	1700	1600	1600	00000 18000 18000	1800 1800	1500	1200		1500 1500 1500	20000000000000000000000000000000000000	25555555555555555555555555555555555555	1500	1500	1500
	Fouring Temp. F	3000	2800	5600	5450	86222 86236 8636 8636 8636 8636 8636 863	2950	5950	% % % %		2020 2020 3030 3030 3030 3030 3030 3030	28888888888888888888888888888888888888	\$\$2222222 \$\$7\$\$\$\$\$\$\$\$\$\$\$\$\$\$\$	2850	2850	2850
	Other	Mischmetal- 0.010 \$ 0.014	S 0.006 B 0.776 Mischmetal- 0.010		S 0.0021 B 0.017	1111	11	ł	111		111	1111111111111	11111111111111	;	i	;
	ි	2.38	1.77		1.80	1111		;	111		111			ł	ļ	!
	5	!	0.248		0.025		il	i	111		88	66	111111111111111111111111111111111111111	i	ļ	:
YSES	퇣	0.14	0.996	0.140	0.0100	1111		1	111		95.1	??!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!!		ł	ł	i
DE ANA	31	0.79	0.015	96.	0.150		11	i	111	a 1.	0.17	00 PH 1 1 1 1 1 1 1 1 1		į	1	;
OM CHAF SIS)	Pe e	1.18	0.122	1.180	0.120	11138	1.38	1.11		material.	15.1	11.52	111111111111111111111111111111111111111	# I	1.27	1.08
ATLD PR	11	1.13	1.006		1.010	0.82	0.82	12.0	0.57	ceramic	9.00	9.99.	:::::::::::::::::::::::::::::::::::::::	0.5	9	0.71
CALCUL	v	0.13	0.140	0.130	0,140	0.13	0.13	1.76	0.13	with	0.16 0.16 1.56	00000000000000000000000000000000000000	00000000004 20000000000004 200000000000	2,96	0.13	0,12
COMPOSITIONS CALCULATAD FROM CHARGE ANALYSES (WEIGHT PERCENT BASIS)	Zr		0.210	0.010	0.120	8888	9.0	0.07	1.02	s filled	888	888888888888	988888888888	0.05	1	;
T COMPO	>		1	i	1	2.23	2.51	i	2.55	, sheets	111	์ เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียนนายนาย เชียน เช เ เ เ เ เ เ เ เ เ เ เ เ เ เ เ เ เ เ	. 18888888888888	;	:	!
MELT	Š	4.82	4.36	8.	4.50	3.600	3.66	3.18	2.52 2.52	turned,	44.6	##55888888888 ##55888888888888888888888	44444444444444444444444444444444444444	2,40	8.00	3.15
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	A1	6.28	5.58	6.38	5.60	7.01 4.92 4.92	6.99	A110y	3.58 3.37	11 furna	Alloy 4 5.80 1 4.90	<i>~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~</i>	00000000000004 000000000004 0000000000	3.23	21.53	31.15
	Ç	13.32	13.95	13.32	14.00	6.02 6.02 6.00 10.71	6.00	Base 9.29	6.10 7.36	on until	Base A1: 12.64 12.64 10.61	\$3888888888 \$4888888888 \$488888888888888	######################################	17.52	10.53	9.53
	Ta			2.38	.,	8.05 8.02 16.26	16.26	Mickel 26.52	8.12 8.12 40.56	as left		ຑຑຓຓຓຓຓຓຓຓຓຓ ໞຆຓຑຨຨຨຆຆຨຨຨ	1043888888888888888888888888888888888888	_		1.69
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	MELT ALLOY			QI	-1	NASA NASA NASA INCO 713-C			NASA NASA NASA INCO 713-C	76 INCO 713-C	1000 713-C INCO 713-C INCO 713-C INCO 713-C	INCO 713-C NKSA NKSA NKSA NKSA NKSA NKSA NKSA NKSA	NASA NASA NASA NASA NASA NASA NASA NASA	ing:	A1 Mod	11 INCO 713C
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APPENDIX D (CONTINUED)

	Remarks	÷	i	ä	ė.	ď	ė	ď					j.	j.	•	•	d.	•	p.d.e.	b.d.	•	ۀ.	ړ.	<u>،</u>	 	ن	۵	ڼ	i		i	i			•
	Wold Temp. F	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	1500	R. T.	R.T.	1600	1600	1600	1600	1500	1500	1500	1500	1500	1500	1500	1500	1800	1800	1800	1500	1500	1500	1500	1200	002
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	Pouring Temp F	8 8 8	3000	2850	865 0	2850	2850	2650	8950	90.90	282	88	ł	88	865	98 20	28	8	80	80			88	8	88	80	88	2300	2300	8	8	8	8	8	3000
	Other	;	!	;	;	;	;	;	ł	i	ļ	}	:	8 0.010 S 0.0005	B 0.01	B 0.01	B 0.01	B 0.01	B 0.01 Mischmetal 0.3	B 0.01	B 0.01	8 0.005 B 0.010	8 0.005 0.005	0000	Mischmetal 0.3	8 0.005 0.005	8 0.005 0.005	0000	8 0.005 B 0.010	\$ 0.005	0.005	8 0.005 0.005	000	0,010	0.00 0.00 0.00 0.00
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22	£	1	;	i	ļ	:	;	1	;	i	;	ļ	;	0,060	!	;	ł	į	i	ŀ	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	o. 10	0.10	0.10
AKALYS	SI		1	ł	:	;	ì	1		1	i	i	;	0.084	į	;	ļ	1	ļ	į	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14	0.14
CHARGE	•	1,33	1,22	1.24	96.0	1.31	1,30	1.39	988	0.88	;	ł	48.0	1.404	0.02	0.02	98.0	98.0	0.85	98.0	1.90	1.90	1.90	1.90	1.92	1.90	1.9	1,90	1.90	1.90	7.8	8.	8.1	2.8	1.91
COMPOSITIONS CALCULATED PROM CHARGE ANALYSES (WEIGHT PERCENT BASIS)	11	98.0	15.27	92.0	9.0	7.43	0.85	5.29	0.56	0.57	}	ł	0.50	0.490	1	ł	ł	1	i	;	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83	0.83
LCULATI BRCENT	υ	8.	5.8	3.78	2.99	1.51	3.8	0.14	3.83	3.83	0.19	0.19	2.96	060.0	60.0	60.0	60.0	6.0	0.18	0.19	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0,15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
TONS CA	Zr	8.06	90.0	0.065	0.059	990.0	0.065	4690.0	0.052	0.054	%.	2.0	0.050	450.0	į	:	0.1	1,0	- [1.0	90.0	90.0	90.0	90.0	90.08	90.0	90.0	90.0	90.0	90.0	90.0	90.0	97.0	97.0	91.0
TI SOUTHO (V)	>	1		;	1	ł	÷	ł		1	2.50	2.50	}	ł	2.5	2.5	2.5	2.5	2.5	2.6	1	1	ł	1	;	ł	ł	i	}	ł	;	ł	ł	;	;
MELT C	Mo	3.86	3.48	18.22	2.42	3.81	3.78	4.03	2.52	2.52	00.4	8.4	2,40	2.50	0.4	0.4	0.4	0.4	0.4	4.1	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17	4.17
	3	1	:	!	21.00	1	6.52	1	20.92	8.8	8.	00.4	21.0	51.6	5.0	0.4	0.4	0.4	5.0	4.1	ļ	į	1	į	;		1	;	;	;	!	ł	ł	ł	!
	V	8.8	. 70	1.17	3.81	5.13	2.08	10.28	3.38	3.38	6.02	6.02	3.23	3.42	0.9	0.9	0.9	6,1	0.9	6.1	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70	5.70
		11.39	10.18	10.32	18.77	14.52		11,88	A1103	17.65			17.52	18.20						7.1	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12.70	12,70	12,70	12.70
	ę,	2,07	1.87	1.89 10	1.51 18	2.04 14	3.06 1:	2,15	8		A110y 99 69.	99. 99.	A110y	17 1	. 6	6	.1 7	.1 7	9 6.7	8.2 7	2.15 1	2.15 1	2.15	2,15 1	2.15 1	2.45 1	2.15 1	2.15 1	2,15 1	2,15 1	2.15 1	2.15 1	2.15 1	2.15 1	2.15
	Ta	64.49 2.		58.52 1.					Non Nickel B 41.84 8.03	.82 8.	1 Base	34 A	1 Base 1.9 10	8	.2 7	0	9 9	8 0.	66.5 7		Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bal. 2	Bel. 2	72.18 2	72.18 2	
	INCO N1 713-C	₹ -			- 47		88.01 63	93.75 67	- W	- 41	Non Nickel Base Alloy 67.34 7.99 6.97	n Nicke	n Micke	3	<i>-</i> 9	. 6	ري. ا	. 38	·8		99.96 B	100 Be	100 Be	100 Be	99.67 Be	100 Bg	100 Bg	100 Be	100 Br	100	100	100	7 6.66		7 8.66
	7.E	1	i	i	1	-			;	ע ע ג	8 !	2 ¦	2% 2%	88	ł	1	;	1	-	i		20	01	10	8		20	10	01	7	Ä	ä	8	83	86
	ALLOY	INCO 713C	Zr-C Mod INCO 713C	T1-C Mod INCO 713C	Mo-C Mod INCO 713C	Cr-W-C Mod INCO 713C	Cr-T1-C Mod INCO 713C	Cb-Ta-M-C M	55 INCO 713C	Ta-C-Cr-W Mod 457 INCO 713C Ta-C-Cr-W Mod	NASA	460-461 462 NASA	120 12V 429	LTV	NASA Mod	NASA Mod	NASA Mod	NASA Mod	NASA Mod	NASA Mod	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	INCO 713C	13C 713C	13c 713c	100 713c	INCO 7130 High Zr.B
	MELT NO.	432	433	4 34	435	436	#37	£38	4. 2.0.7.	457	4 4 4 4 4 4	400	463-5 527	5.28	529	530	531	535	533	534	535	536	537	538	539	540	541	545	543	544	545	946	247	548	5#6

APPENDIX D (CONTINUED)

	Remarks	в.е.		a.e.		
	Mold Temp. F	1200	1200	1200	1200	
	Pouring Temp. *P	2750	2800	2800	2800	
	Other	s 0.005	0000	8 0.040 0.005 0.005	ы м и 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.	
	ಕ	1	;	-	!	
	8	0.05	0.05	0.05	0.05	
2	Ę	0,10	0.10	0.10	0.10	
ANALYS	SI	0.14	0.14	0.14	0.14	
CHARGE	e.	1.94	₽.1	1.92	1.91	
ED FROM	E	0.82	0.82	0.81	0.80	
ALCULATI PERCENT	0	0.15	0.15	0.15	0.15	
MELT COMPOSITIONS CALCULATED FROM CHARGE ANALYSES (WEIGHT PERCENT BASIS)	72	0.18	90.0	0,08	90.0	
COMPOSI	>	ł	ł	-	}	
MELT	Š	5.8	5.08	5.04	5.01	
	3	i	;	0.37	1.93	
	A1	6.10	6,10	5.56	2,52	
	ç	12.49	12.49	12,39	12.30	
	Та	2,60	2.60	2.58	2.57	
	N1		70.97	20.42	69.88	
	1NC0 713C	97.18	97.8	98.19	97.53	
	ALLOX	INCO 713C H1gh 97.8	INCO 713C High	INCO 713C	INCO 713C Mod	
	MELT NO.	550	551	555	553 ₩.₩	

a. Silica Mold,
b. Zirconta Mold,
c. Alumina Mold,
d. Iron added with the vanadium as ferro vanadium,
e. Iron added with the boron as ferro boron,
f. Metted in graphite orucible,
f. Metted 359 thru 465 melted in Ril 152 curcible,
h. Metts 457 thru 462 melted in zirconta crucible,
h. Metts 529 thru 553 melted in Magnorite X orucible,

APPENDIX E

VENDOR TYPICAL CHEMICAL AMALYSIS FOR MELT CHARGES

WEIGHT PERCENT

	సై	25				5 02, 0.04 H2	B, 20 ppm Co, ppm M ₂	1.0 volatile matter, 0.5 moisture	0.03 Cu, 0.025 S, 0.03 P, 0.15 Mn, 0.04 As 0.15 Co, 0.025 Ca
OTHER	0.10 Mn, 0.25 Cu	0.10 Mn, 0.05 Cu			0.10 P, 0.03 S	0.002 Hz, 0.06 Oz, 0.04 Hz	2.5 Mf, 1 ppm B, 20 ppm Co, 50 ppm Pb, 80 ppm M ₂	1.0 volatile m	0.03 Cu, 0.025 0.04 As 0.15 C
S1	0.17	0.1 th		.0003 .000 4	8		00 Edd		1.40
Fe	0.09 0.16 0.97 1.51	1.8		.0003	.25		.15		23.8
Ţ	0.97	0.15 0.83					00 E		
ບ	91.0	0.15			55	, too		88.5	.15
73	0.09	90.0					Bel.		
>	•	ı				% 8.	50 ppm		70.5
Š	4.32	4.17		цю. 6.66	98.0		50 Ppm		0.08
*	•	•		9.9			50 100 100 100 100 100 100 100 100 100 1		
¥	12.64 5.80	12.70 5.70							0.80
The Cr Al W No V Zr C		12.70		.00			0.03		0.10
Ę	2.30	2.15							
Mi	71.68	Bel.	available	.0005			0.0		0.01
PRINCIPAL METAL	INCO 713C	INCO 713C	Ta, Or, Al, Ti unavai	>	o x	>	ä	U	Ferrovanadium

APPENDIX F

NICKEL ALLOY ROLLING DATA ROLLED AT METALS & CONTROLS

REMARKS	Sheet annealed (between every 3 passes) at 2150°F for 1/2 hr. air cooled	Annealed at 2000°F for 1-1/2 hrs. before hot rolling.	Annealed at 2150°F for 1-1/2 hrs. before hot rolling	Surface belt sanded before start annealed 9 hrs. at 2150°F after 4th pass air cooled.	Surface belt sanded before start no anneal. Badly cracked throughout sheet.	Same as #77	Annealed 1-1/2 hr at 2150, water quenched after 4th pass Annealed 1/2 hr at 2150, water quenched after 6th to 8th pass Annealed 1/2 hr at 2150 air cooled after 9th pass	Annealed 1/2 hr. at 2150, air coolad after 3rd and 4th pass. Cut in two after 4th pass, 1/2 continued on 7" mill to .072" other half rollad on 20" mill to .083"	All passes except 3rd done on 7" 2 high mill, amosaled 1/2 hr. at 2150, water quenched after 2nd pass. Amosaled 1/2 hr. at 2150, air cooled after 3rd pass cut in two, both halves rolled on 7" 2 high mill
FINAL SHEET CONDITION	Badly Cracked	Badly Cracked	Badly Cracked	Badly Cracked	1/4" deep edge cracks	1/4" deep edge cracks	Bedly Cracked	Bedly Cracked	Badly Gracked
MAX.LOAD PER IN. OF WIDTH (pounds)	:	!	1	ł	1	;	;	1	1
MAXIMUM IOAD (pounds)	1	;	:	1	;	;	•	1	;
AVERAGE REDUCTION PER PASS (.001")	3.0	3.5	0.4	0.4	0.9	3.0	9.0	3.5	ę. S
WIDTH (1n.)	;	;	1	;	;	:	1	:	:
FINAL THICK- NESS (.001")	\$ 88	74 to 82	85 82 84	72 to 75	86 to 87	8 8 8	61 to 63	72 to 1/2 83-86 on other	75-78 on 1/2 73-75 on other
INTITAL THICK- NESS (.001")	100 to	96 to 111	97 to 116	97 to 106	011	95 to	98 to	95 to 102	101 106
MIL	7"Dia. 2 high	7"Dia. 2 high	7"Dia. 2 high	20"Dia. 2 high	7"Dia. 2 high	7"Dia. 2 high	7"bis. 2 high for first 1/2, 20" bis. 2 high last	7"Dia 20 bigh & 20 bigh 2 bigh	7"Dia. 2 high & 20"Dia. 2 high
ROLLING TEMP. (*P)	Room	Room on 4 passes 1670 on last 2	Seme as #74	Room	Room	Room	Room	Room	Room
MELT.	914	416	419	114	388	1	419	75°	524
ALLOY	NASA	NASA	KASA	MASA	Ta-c modi- fied INCO 713C	7130	MASA	IIASA	KASA
ROLL	73	4	75	92	F	<u>8</u> 2	6	&	6

APPENDIX F (Continued)

HICKEL ALLOY ROLLING DATA ROLLED AT VOUGHT

RIBARITS		Temp. increased to 1800 on 8th pass (2" L.D. glo-bar furnace used for roll numbers 82 to 110 inclusive) Temp. increased to 1800 on 11th pass. Temp. decreased to 1800 on 15th pass.	Twup. at 1950 for pass \$9-11,16,17; Twup. at 2000 for pass \$6-8,15; Twup. at 2050 for pass \$12-1k; Twup. at 2150 for pass \$1,3; Twup. at 2250 for pass \$1,3; Twup. at 2250 for pass \$18-21; Furnace soaking time varied from 5 to 60 minutes	Same as 83-1	(Two pieces) one specimen stopped on 5th pass due to lack of room in furname. Twmp, at 2000 for passes \$12.7 Twmp, at 2005 for passes \$13.7 Twmp, at 2100 for passes \$1-12, 14-18 Soaking time varied from 5 to 30 min.	Original condition of surface, poor	Final surface of specimen in good condition, very slightly oxidized	Specimen encapsulated in sheet from malt 459 for passes \$1.4; recovered with stainless steel for rest of test. Stainless steel elongated more than specimen, thereby breaking up sheet.
FIELD SELECT COMMITTION		1/8 to 3/16" deep edge cracks	A few 3/8" deep edge crecks	Sene as 83-1	About the same as 83-1	Badly Crecked	1/32" deep edge crecks	Com- pletely cracked up
MAX.IOAD PER IN. OF WIDER (pounds)		009 , 84	73,900	73,900	005°C01	:	I	ŀ
MAXIDEIM IGAD (pounds)		99,600	73,900	73,900	005°C04	:	1	1
AVERAGE REDUCTION PER PASS (.001")	SEETH THICKNESS (.001") PER PASS		6 for 1/2, 3 for other half of two	6 for 1/2 3 for other half of run	v	9	v	vo
WIDE (10.)		H	н	н	-	т	1	1
TITAL THICK- HESS (.001")		37 to	37 to	36 36 36	90-112 on one 40 on other	83-86	:	57. 52
INITIAL THICK- HESS (.001")		81	ដូដ	% £11	94-112 on one 40 on other	9	110 111 12	99 ts 110
HEIT		6"bie. 2 bigh 5 m.P.		Same	Same	Same	Seme	Seme
ROLLING TIME. (*P)		1700, 1800 1880 800 re-	2000 2000 2000 2000 2000 2000 2000 200	Same as 83-1	2000 2050 2100 2150	3000	2500	880
X		8	654	3 3	8	8 9	1,57	જુ
ALLOX		174-5 10041- 11000 7130	KABA	INSA	KASA	MASA	Pa-C- Cr-t- Modi- ried 7130	HASA
ROLL		8	83-1	83-2	ಹೆ	85	8	84

APPENDIX F (Continued)

REMARKS	Chilling of sheet caused by drive mechanism slipping.	Used for tensile bar.	Gracks widened but did not deepen at end of test, used for tensile test.	.018" shim on pass #2006" on others	Sheet looked in good shape		.003" shim on passes #8-17, .006" shim on passes 3-7, .009 on passes #28 18	Sheet annealed at 2250 for 10 minutes after 9th pass after which it broke up	Specimen bowed during rolling due to unequal tension on working rolls-caused alipping.	Specimen bowed badly causing cracking	1/2 of a failed tensile bar surface in good condition	Sheet looked good at end, very little bowing, crack widened but not deepened
FINAL SEET CONDITION	Visible surface cracks	1/32" edge cracks	1/32" edge cracks	Com- pletely cracked	1/16" deep edge cracks	Same as 92	Same as 92	Badly	Bedly Gracked	Badly	1/64" edge crecks	3/16" deep edge cracks
MAX LIDAD PER IN. OF WIDTH (pounds)	1	;	;	;	1	:	:	:	!	;	:	;
MAXIMIM LOAD (pounds)	1	!	:	:	:	:	:	ł	:	:	;	ţ
SHIDM THICKNESS H PER PASS (.001")	9	m	m	6 18	m	m	3,6,9	m	m	9	m	e
WIDTH (in.)			•	•			ı			1	1	ı
FINAL THICK- NESS (.001")	40 to	12	82.88 S	90 to	ß	58 60 50	54		85	- 80 €	15 to 16	12 16
INITIAL THICK- NESS (.001")	101-	102- 115	114 - 115	aa	115 - 125	107 - 113	2 <u>7</u>	011	95 to 103	97 to 112	878 5	901 011
MIL	Seme	Seme	Same	Same	Same	Same	Same	Same	1-1/2" Ma. 4 Hgh 5 H.P.	Seme	Same	Same
ROLLING TEMP. (*F)	2100	1500	1750	3000	1850	1650	1750	1750	1750	1825	1825	888
MELT.	154	Z9 1	8 9	89	8	3	8	8 9	ł25	39	3 9	₫
ALLOY	Ta-C Cr-W Modi- fled INCO 713c	KASA	KASA	NASA	MASA	MASA	KASA	RASA	MASA	KASA	HASA	713c
ROIL	88	86	8	ደ	84	æ	ま	82	%	8	98 contin- uation of 90	8

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RIBARKS	Working roll size too small for thichness of sheet.	1/2 of failed tensile bar. Specimen in good condition at .030" thickness.	Other 1/2 of tensile bar from roll #66 final surface condition good	Tensile bar that broke while drilling final surface, good	Other half of tensile bar that was run on run 98, good surface	Mill was shimmed every other pass only. Slightly oxidized surface.	Same as #105	Non-nickel base rolling	Same as roll #105	Same as 105
FIRAL SHEET CONDITION	Badly Cracked	Bedly Cracked	1/32" edge crecks	1/16" edge crecks	1/32"	1/8" edge cracks	1/8" edge cracks		1/8" edge crecks	1/16" deep edge cracks
MAX. LOAD FER IN. OF WIDTH (pounds)	1	1	1	ı	:	:	:		:	:
MAXIDEDM LOAD (pounds)	1	1	1	ı	:	1	i		ŀ	1
SHIPH THICKNESS TH PER PASS (.001")	m	m	m	m	m	3 See ra- marks	8 3		/2 3 See re- marks	m
MIDTA (.at.)	•	±√2 3/4 3/7	3/4-	3/4-	3/4	1	1-1/2		1-1/2	1
THICK- THICK- KESS (.001")	7 02	15	88 88 5	& % %	8 8 5	53 57 50	84.85 5			3 54 4
THICK- THICK- NESS (.001")	109 -	48 to	47 to	b3 t4 t4	\$ 8	103- 11-	38 102 23		3 2 2 1 2 3 3 4 3 4	% to
MIL	S	Seme	Seme	Seme	Seme	6"bia 2 high 5 H.P.	Seme	•	Seme	Seme
ROLLING TIME. (*P)	2100	2100	2100	2100	1825	1825	1825	•	1825	1825
Menty.	#21	254	154	T54	8 3	¥18	¥25	,	41.8	61.4
ALLOY	Ta-C- Cr-W Modi- fied IMCO 713c	Ta-C Gr-W Modi- fied IMCO 713c	Ta-C- Cr-W Modi- fied INCO 713c	Ta -C - Cr -W Modi- fied INCO 713c	KASA	HABA	KASA		RASA	KASA
ROLL #	100	101 contin- uation of 88	102 contin- uation of 88	103 contin- uation of 88	104 contin- uation of 90	205	106	701	108	607

	R BHA RUS	.0015" shims for passes #12-15,17,20,22,25 26-28 .003" shims for passes #2-5,8,11,32,34-27. Other passes had no new shims Continued as #114.	Non-nickel base rolling	.003 shim on passes #2-6, 9-15, 17-23, 25,27,30,36,39. Bo new shims on other passes (large glo-bar furnace used from here to end)	2 high mill with power increased to 30 HP .003" shim on passes \$1-4 .006" shim on passes \$5-11	.006" shim on passes 1-18 on 6" Dia.,2 high mill; .003" shim on passes 19-38 on 1-1/2" dia., 4 high mill original width 4" then specimen was cut into 1" wide strips for continuation on 4 high mill;	Non-nickel base rolling	2 high & 4 high mill used. Changed from 2 to 4 high at pass \$29. Rolled 5-1/8" width at 1225°F on passes 1-7. Rolled 2 pieces 2-1/2" width at 2015 on passes 8-48, for tensile bars	Rolling temperature 1925°F for passes 1-7, 1875°F for 8-26. Sheet bowed considerably due to variation in thickness	No new shims on passes \$26-26, 32-34, 36, all others .003 shims. 1/3 of sheet completely cracked up by pass \$10 .herefore 2/3 of sheet continued	Rolled on 2 high mill at 1875°F during passes 1-27. Nost of sheet then used for tensile specimens. Remainder continued on high mill at 2025, passes 28-43	Rolled on 2 high mill at 1875°F during passes 1-36, then trimmed to 3-3/4" width and continued on 4 high at 2010 for passes 37-43. Tump. releed to 2200 for passes 44-54.
	FINAL SHEET CONDITION	1/8" deep edge cracks		1/8" deep edge cracks	1/8" deep edge cracks	1/8" edge cracks		1/8" edge cracks	Edge & interior crecks	1/8" edge crecks	3/16" deep edge crecks	1/8"- 3/16" edge crecks
	MAX. LOAD PER IN. OF WIDTH (pounds)	:		24,000	:	26,000		13,600	008,51	11,500	:	:
APPENDIX F (Continued)	MAXIMIM LOAD (pounds)	ŀ		52,000	90,00	106,000		45,000	, 11, 800	₩,500	:	i
APPENDIX F	SHIPM THICKNESS FER PASS (.001")	0,1.5, 3.0 See re-		O,3 See re- merks	3,6 See re- marks	3,6 See re- marks		m	e	0,3 See re- marks	m	m
	WIDIN (1n.)			2-1/8	•	See re- nerits		See re: marks	3-1/2	4-3/4	2-1/2	4-1/2 at start
	FINAL THICK- NESS (.001")	83		45 to 46	51 to	15 to		21-	386 3	አ ሄ ኔ	4 <u>2</u> 5	88
	INTITAL THICK- NESS (.001")	112-		108 108 10	83	\$ &		ង្	-101 921	. 101 101	2 103 103 103	98 100 201
	MIL	Same	1	Same	See re- narks	See re- marks	•	See re- marks	2 h1gh	į	See re-	See re-
	ROLLING TEMP (*F)	1825		2100 See re- marks	2130	80%		See re-	See re-	1925	See re- marks	See re-
	MELT.	415		FO5	415	4.18	•	387	3	5 24	1	425
	ALLOY	HASA	,	713c	MASA	HASA		KASA	MASA	HABA	7136	HASA
	ROLL	110	111	112 # 113	114 contin- uation of 110	115	116 118	611	8	ផ្ដ	ឌ្ន	ध्य

APPENDIX F (Continued)

RBARIGS	Nolled on 2 high will at 1925" during passes 1-36 then cut into 3 pieces, 2-1/2", 2-3/6" & 3-3/ $^{\mu}$ " wide and continued at 2020" on 4 high mill	Two sheets rolled on 2 high mill at 1875° during passes 1-30 then part cut into tensile bars. Remainder continued at 2015° on 4 high mill for passes 31-44. Part rolled cold as roll #32.	Sheet cut into tensile bars, rest con- tinued as roll 131	Sheet cut into tensile bars	3" wide sheet rolled on 2 high mill during passes 1-26 then trimmed into several pieces ranging from 1" to 3" wide and continued on 4 high mill.	Some sheet to tenaile tests	2 sheets, cracking due to variation in thickness	Bowing caused by different tension in rolling mill drive chain, causing cracking	Annealed at 2100°F for 1 hr, air cooled after lith pass	Sheet from previous hot rolling on 2 high mill	No significant new cracks only once from 2 high rolling opening up
FIRAL SHEET CONDITION	1/8" edge crecks	1/8" edge cracks	1/4" deep edge cracks	3/16" deep edge cracks	1/8" deep edge cracks	3/16" deep edge cracks	Badly	Badly Cracked & Bowed	Badly Cracked & Bowed	Bedly	Cracked
MAX.IOAD PER IN. OF WINTH (pounds)	í	13,800	:	:	:	:	1	9,800	1	:	:
MAXIMEM IOAD (pounds)	1	38,000 4 da h1da	:	;	ŀ	:	ł	27,000	:	:	i
SHIDK THICKNESS PER PASS (.00.")	m	m	m	m	m	m	m	e	m	m	m
WIDIN (in.	6 at start	2-3/4	2-3/4	2-3/8	3 See re- narks	2-3/8	2-1/4	2-3/4	1	1/5	a
THICK- THICK- HESS (.001")	ద్ద ష క	31 to	33 23 33	2 2 2 2	3 ನ ಣ	8 8 3	% % %	18 16 16	88 &	38 38 38 39	ង
INTIAL THICK- HEBS (,001")	38 112 2	95 to 108	94 to 108	88 106 53	95 to 110	98- 106	82 to 105	73 73 53	74 to	1,47 1,68 1,0	48 3
TIM	See re- marks	See I'e- Marits	2 high	Seme	See re- marks	2 h1gh	2 high	पृष्ठीप १	कीप १	4914 1	t pret
ROLLING TING. (*)	See re- marks	See re- marks	1875	3000	800	3000	88	2000	Room	Room	2100
*	8T.4	385	₹ 7	7	₹. 1	1 04	86	なって	385	†	\$
ALLOY	KASA	NASA	KASA	13C0 713c	IIASA	13c	Ta-c Modi- fied INCO 713c	MASA	KASA	713c	KASA
ROLL	द्य	521	9टा	121	821	627	95 95	131 126 con- timed	132 125 cm- timed	£13	13th 120 con- timed

	REMARKS	All through sheet at grain boundries	Ubcracked area from 2 high rolling	Refer- data for 4 high mill, roll 122.	Refer to data for 4 high mill, roll 119		Refer- data for 4 high mill, roll 128	Refer to data for 4 high mill, roll 115	Refer to data for & high mill, roll 124	Refer to data for 4 high mill, roll 123	Refer to data for 4 high mill, roll 128	Refer to data for 4 high mill, roll 117	Refer to data for 4 high mill, roll 125	Refer to data for 4 high mill, roll 128	Refer to data for 4 high mill, roll 119	Refer to data for 4 high mill, roll	Refer to data for 4 high mill, roll 115	Non-nickel base rolling	Fart of riser screp from casting surface ground to parallel faces, no zyglo defects
	FINAL SHEET CONDITION	Bedly Cracked	Some edge cracks			1/16" edge cracks													Com- pletely broken up
	MAX LOAD PER IN. OF WIDTH (pounds)	ť	1																43,300
Continued)	MAXIMUM IDAD (pounds)	ŀ	:																65,000
APPENDIX F (Continued)	SHITM THICKNESS FIER PASS (.001")	m	m			m													6,12,15 30
•	WIDIN (in.)	1-1/4	7			2-3/8													1-1/2
	FINAL THICK- MESS (.001")	8	% ₹ \$			ଝ													ま
	INITIAL THICK- NESS (.001")	ខ្លុំដ	₹ &			52 ts													4. 2. 2. 3.
	MIL	popu †	4914 7	prese	t pict	4 h1gh	प्रवीप १	ф. 1	प्रकाप १	t pigh	popu †	pring †	4 H.gh	ф 1164	4 b164	प्र क ीम १	propre	!	2 high
	ROLLING TEMP. ('F)	2100	3000			2025													9000
	*	530	Ž,	₫	387	†	454	418	418	425	727	397	385	₹ <u>₹</u>	382	385	61.4		
	ALLOY	KASA	KASA	7130	NASA	7130	MASA	MASA	MASA	NASA	NASA	NASA	MASA	KASA	KASA	KASA	ILASA		EASA
	ROLL	135	136,137 130 con- timed	138 • 139	140	141 contin- uation of 129	142 2 143	141 2 145	146 147 148	149	150	151	152	153	<u>국</u>	156	151	158 159,	191

	REMARKS	Part of a 3" consummbly are-melted ingot, transverse alice; .003" shim on passes #10-33; .006" shim on passes #1-9	Non-nickel base rolling	Rolled sheet cut into tensile specimens	Sheet cut into tensile specimens	Hon-mickel base rolling	Surface crack appeared on pass #16 .0016" shim on passes #31-39; .003" on others. Crack due to variation in thickness	Non-mickel base rolling	large grain size sheet plus variation in thickness caused breskup.	Same as 175	Cracks appeared at grain boundries after macro etching.	Speet from 530 started on pass \$1 then one from 536 started on pass \$20
	FINAL SHEET COMPITION	Com- pletely broken up		1/8" edge cracks	1/16" edge crecks		1/8" edge cracks & surface cracks		Com- pletely crecked	Com- pletely crecked up	Buge & surface crecks	1/8" edge cracks
	MAX LIDAD PER IN. OF WIDER (Tremds)	24,000		1	1				:	16,600	1	1
APPENDIX F (Continued)	MAXIDADM	30,000		:	:				!	%°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°°	:	1
APPENDIX 1	SHITM THICKNESS PER PASS	(.001") 3,6		m	m		See re- marks 1.5,3		m	m	m	m
	WIDIA	(in.) 1-1/4		2-1/8	2-1/4		m		m	m	N	'
	PUM. THICK-	î.		&8 3	56 to 59		55 57 50		175- 176	175	7. 13.	3 39 33 50 50 50 50 50 50 50 50 50 50 50 50 50
	INITIAL THICK-	(.001") -244 -66	ļ	105-	, 11, 12, 13, 11, 11, 11, 11, 11, 11, 11, 11, 11		88 23 33		197-	184-	101- 205	99-103 \$8-60
	į	म्ब र		प्रकाप ट	पूर्व र		2 h@		15 PT 2	2 संख्	क्रीम ट	है इस इ
	9077	13 (14) 13 (14)		1950	1950		800		2100	2100	2100	88
	MELT ROI		ra- merks	61 524	3 6 5		£19 2		536	535	537	537 538 966 166
					KASA 3		MASA.		1700 713°c	00	13C0	7130
		ALLOY 162 KASA		163, 164, 165			168, 169, 171		. ,511 151 1 51	176	111	178

APPENDIX F (Continued)

REMARKS	Initial condition of surfaces was poor	Initial condition of surfaces was poor	Initial condition of surfaces and thickness variation poor	Rejected sheet (holes) yet rolled well. Temp.at 2000°F during passes 1-18, 2100°F for remainder	Temp. increased to 2210°F on 5th pass	Since distance between 4 high bearings was 16" some difficulty was encountered in placing sheet in rolls, causing cracks	Sheets from roll 1/79. New cracks aggested in area which was not cracked in roll 1/79	Considerable bowing throughout run	X-ray segregation & surface pores at start	Cracks in surface at grain boundaries	large thickness variation contributed to sheet bowing throughout run
FINAL SHEET CONDITION	Gracked	Gracked up	Crecked	1/8" edge cracks	1/2" edge cracks	1/2"al" edge cracks	Cracked	1/8" deep cracks	Bro ken up	Burface & edge cracks	Cracked
MAX LIOAD PER IN. OF WIDTH (pounds)	ı	:	:	:	:	:	:			:	1
MAXIMIM IOAD (pounds)	1	1	:	ŀ	1	:	:			1	1
SHIDM THICKNESS WIDTH PER PASS (1n.) (.001")	1 8 e 3	38.	m I		10-1 / 4 3	13-5/8 6	m	es a	m	m I	2-3/4 3
THICK- HESS (.001")	52 to 55	1	•	ያ ጽ ≵	₹ 20 20	16 to 51	5 13 14 15	₹. 3	47-52	3 3 3	₹ &
INTTIAL THICK- NESS (.001")	105- 110	98 to 80 to 75-71	94-110 89-92 41-90	103-	8 K	\$ 1 .82	88 3 3		# 288 73	88 3	52-92, 65-106
K	2 महिम	प्रक्रम इ	2 high	2 भाकुम	property 4	19 714 1 7	प्र व ीम १	4974 1	₽	कीम १	क्ष्म ट
ROLLING TEMP. (*F)	3000	8	5000	2000	22.20	2500	8050	8000	8000	1900	8000
MA THE	537	537 & 539	£•£	105		1 05	537	533 539	₹	₹	\$
ALLOY	INCO 713c Two pteces	IMCO 713c four pieces	INCO 713c three pieces	T13c	13c	11800 713c	113c 113c four pdeces	77.30	TIECO TIECO Trees prieces	INCO 713c two pdeces	713c 713c pdeces
ROLL	179	981	181	182	183	184	185 contin- uation 179	186 contin- ustion of 180	181	8 8	681

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PARIMITY P
DOMESTY P

RIBIARICS	Non-nickel base rolling	Part of sheet that cracked up was X-ray rejected area	Same as 191 (two pieces)	Same as 189 & 191 (three pieces)	Same as 189 & 191 plus bowing	Same as 189 & 191	Non-mickel base rolling	Annealed at 2200°F for 15 hours after #7 pass; thickness variation caused cracking	Senne as 200	Same as 200	Rolled at 2050 for passes \$1-34, 1900 for remainder, thickness variation caused cracking	Mon-nickel base rolling	Rolled at 1900°F for pass 1-33 then temp. increased in 2000°F			
FIRAL SHIEF CONDITION		See remarks	See remarks								Completely Cracked		Cracked com- pletely	Com- pletely cracked		Surface, edge cracks
MAX . LOAD PER LH. OF WILKH (pounds)		:	:								6,200	5,400	18,200	13,600		14,500
MAXIDAM LOAD (pounds)		1	:								50,000	43,000	100,000	61,000		87,000
SEEDA THUCKATERS PER PASS (.001")		m	m	m	m	m	m	m	m		m	m	m	m		e
WIDIN (in.)		1-3/4	2-1/2	2-1/2	2-1/5	2-1/2	α.	4-1/2	m		60	6 0	5-1/2	4-1/2		v
THAL THUCK- HESS (.001")		9 1 1	₽3- ₽2-	-94 84	& &	52-	43-55 43-55 43-55	48-54	48-52		89-92	89-92	84-₹t	83-87		53-55
INTELAL THEOR- HESS (.001")		95	94-106	67-102 60-97	53-69 88 cest	32-66	45-65 43-60 45-65	57-64	49-64 22-64		92-110	87-109	9 4 -113	140- 172		101 107
MIL		2 संदू	2 116	2 high	प्रधीय ट	2 h1gh	2 high	2 high	2 bigh		प्रकृप ट	2 118	2 घडक	2 h1gh		2 high
ROLLING THEO (*P)		2050	2050	2050	20%	2000	3000	2000	800		2000	3000	800	See re- marks		
Ž.		₹	₹	246	\$	5 <u>4</u> 6	1	₹	\$	-	₹	2 4 5	2 4	¥.		15 8 17 8
ALLOY		7136	773	7130	113c	1MC0 713c	113c	13c	13c		713c	118C0	113c	1MC0 713c		13c
WOLL.	961	161	138	193	\$	195	196	197	198	199	8	1 00	8	803	\$ 2 &	210

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REMARKS	Jammed mill on 37th pass, thickness .068"	hill jammed several times. Thickness variation caused cracts.	Strain recorder failed after pass 18. Thickness variation caused cracks.		One large internal crack due to thickness variation. Piece cut in two after 104,000# load and continued	Rolled at 1950 for passes 1-22 & 29-48, 2000 F on others. Sheet cut in two at pass #28-part not continued. Maximum load per width based on narrower (2 7/8) sheet.		Non-nickel base rolling	One of sheets from 217 cut transversly in half, .003" shims used for passes #1-10, then tapered shim blocks with tightening screws employed	One of two sheets from #214	Old cracks opened up. Will preloaded to 20,000# with tapered shim blocks.
FINAL SHEEF CONDITION	Surface edge cracks	Surface edge cracks	Surface & edge	Surface & Edge cracks	Edge cracks		Edge & surface oracks		1/8" edge cracks	Broken up	Seerensrks
MAX.LOAD PER IN. OF WIDTH (pounds)	16,700	22,500	10,200	20,600	17,600	31,000 See re- marks	19,800		11,100	7,800	10,200
MAXIMUM IOAD (pounds)	100,000	135,000	61,cco See re- marks	67,000	104,000	89,000	154,000		30,500	82,000	102,000
SHILM THICKNESS FER PASS (.001")		m	m	en t	m B	m +	m		See re- Berks	/8 Same as #221	Same as #221
WIDIN (in.)	vo	9	9	3 1/4	5 7/8	5 3/4	7 3/4		2 3/4	10 3/8	10
FINAL THICK- NESS (.001")	55-56	53-55	L9-99	44-64	55-57	45-50 82-85	80-81		19-20	49-50	22-23
INITIAL THICK- NESS (.001")	101	93-120	100-127 101-121	55-63 88 cast	105-119	106-110 g	103-119 128-131		45-50	92-99	80-81
MILL	2 h1gh	2 high	2 high	2 high	2 high	2 high	2 high		4 high	h high	t high
ROLLING TEMP. (*F)	1950	195c	1950	2050	1950	See remarks	548, 1950 549		1950	1950	1950
# # T	747	548	542	543	550	550	548, 549,	:	550	547	240
ALLOY	113c	INCO 713c	INCO 713c two pieces	INCO 713c	INCO 713c	INCO 713c two sheets	INCO 713c two sheets		INCO -713c See re-	INCO -713c See remarks	INCO 713c
TIOE #	211	212 & 213	214	215	216	217	218	219	221 INCO 217 con-713c tinued See re-	222 INCO 214 con-713c tinued See remar	223 218 con tinued

APPENDIX F (Continued)

	RBARKS	Roll dimmeter too small.	Roll dimmeter too small.	Both sheets broten. Foll digneter too small.	Roll dimmeter too small.	Roll dismeter too small.	Cut into tensile bars.	as 23C.		Able two masses at 2100 with uit preloader to 20/000 me with with ablit preloaded to 40,000 Maximum load recorde was that alone 40,000; preload	Rolled 20 to 30 passes at 4.T. with mill preloaded to 40,000. Further preloading caused mill difficulty in starting	Specimen annealed at 2150 for 5 min. between every 5 to 7 passes
	FTHAL SHEET CONDITION	Com- pletely cracked	Com- pletely cracked	Com- pletely cracked	Com- pletely cracked	Com- pletely cracked	1/16" edge cracks	1/16" edge cracks	See remar:s	1/10 edge cracks	1/16 edge cracks	1/16 edge cracks
	MAX.IDAD PER IN. OF WIDIN (pounds)	006 '9	10,100	10,800	8,700	12.000	7,300	ŀ	12,300	13,300	6,700	6,700
	MAXIDEIM 10AD (pounds)	12,000	30,100	19,000	56,000	30,100	22,000	1	80,00C	20,000 See re- marks	Same as 234	Same as 234
HEEDINGE .	THICKNESS PER PASS (.001")	m	m	m	m	æ	Same as 221	Same as 221	9	See re- marks	See re- marks	Same as 235
	(tn.)	1 3/4	۵	1 3/4	m	2 1/2	m	m	6 1/2	1 1/2	m	m
	FIRAL THICK- NESS (.001")		95-97	75-77	ħό	62	25	19-20	30-31	10 I u	7-1/2	5.4 5.
	INTERAL THICK- NESS (.001")	71-81 as cast	106-116	165-111 103-106	104-115	71-17	55-57	45-50	82-85	17-15	16-20	18-20
	MIL	t high	t high	4 nigh	t nigh	4 high	t high	4 high	dgid 4	d hijh	4 nigh	dgin 4
	ROLLING TIME. (**)	1950	1950	1950	1950	1950	1950	1950	1900	See re- marks	R.T.	R.T.
	TI	551	552	553	552	551	550	550	550	550	550	550
	ALLOT	INCO 713c	INCO 713c	INCC 713c two sheets	INCC	IRCO 713c	INCO 713c	INCC 713c two	IECC 713c	INCO	INC0 713c	INCO 713c
	FOLT.	224]	225	226	228	559	230 231 216 con	g.	233 217 con	234 from 232	235	235 from 232

APPENDIX G
TENSILE DATA OF NICKEL ALLOYS
ROOM & 1900^O
LOADING RAIR 16,000 #/1n² per minute

ELONGATION (PERCENT) ROOF 1500*F	1.0 4.0		2.0	1.0 1.0 2.0	1.0	~ o • o	1.0	3.0	1.0	11.5	2.0	1.0 2.0	00
ATE LS STH 1) 1900°F	45,500 45,900 45,4000	24,600	53,200	52,300	53,300	60,100		20,600		19,400	37,300 28,500	37,000 32,900	38,800 38,600
RO	116,200	118,600	onc font	52,600 137,600			111,600		116,200		105,000	127,000 83,400	107,000 95,600
ULTIMATE TENSILE LOAD # OM 1900*F	1260 1265 1650	1050 570 1205	1370	730	940			# 5#		388	1115	1500	1435 1600
ULTII TENS: LOAI ROOM	3010 2830	3590	500	1325 1730	1435	1505	2225		3070		5420 5310	6030 4070	5430 5140
ULTIMATE YIELD STRENGTH (PSI) ROOM Temperature	112,700	111,100		129,700							83,700 115,500	116,100	97,800 85,400
AREA (square inches)	02591 02596 02337 02544	0.000000000000000000000000000000000000	.02574	.01431 .01397 .01334	.01705 .02531	930. 40.	,0199 ⁴	.02202	.02642	.01999	.0516 .0407 .0299 .0478	.04741 .0483 .0405 .0377	.0506 .0537 .0370 .0414
THICKNESS (.001")	100. 100. 92.3 103.1	1000.1	102.9	0.5000 0.5000 0.5000	0,40	116.2	78.1	84.5	105.1	80.3	103.6 80.2 69.7 111.9	98.1 98.8 104.8 96.9	102.2 108.1 98.2 105.4
TEST TEMP. (F)	Room 1900 1900 1900	1000 m 10	1900	1000 1000 1000 1000	1900	1900	ROOM	1900	ROOM	1900	ROOM ROOM 1900 (X) 1900	ROOM 1900 (X) 1900	ROOM 1900 1900 (x)
CONDITION	as cast as cast as cast as cast			<u>a a o ,</u>		as cast	as cast	as cast	as cast	as cast	as cast as cast as cast as cast	as cast as cast as cast as cast	as cast as cast as cast as cast
MELT #	38888 2888 2888 2888 2888 2888 2888 288	- 00 0 0 0 00 0 0 0 00 0 0 0	1 1 6	2000 2000 2000 2000 2000 2000 2000 200	37 4	762	456	426	124	124	2000 8000 8000	2000 2000 2000	2222
ALLOY	NASA NASA NASA NASA NASA	NASA NASA NASA NASA	MASA	NASA NASA NASA	NASA NASA	NASA	Cr modified NASA	Cr modified NASA	Cr-Al modified NASA	Cr-Al modified NASA	Modified NASA Modified NASA Modified NASA Modified NASA	Modified NASA Modified NASA Modified NASA Modified NASA	Modified NASA Modified NASA Modified NASA Modified NASA

					APPENDIX G	APPENDIX G (Continued)						
ALLOY & ROLL NUMBER	MELT NUMBER	CONDITION R	test Temper- Ature	THICK- NESS	AREA	ULTIMATE YIELD	ULTIMATE TENSILE	IATE	ULTIMATE TENSILE STRENGTH	6 3	ELONGATION (PERCENT)	TION
			(_*)	(,001")	(sq. in.)	(PSI) ROOM	(#) 1900(1900°F	(PSI) ROOM	4.006	ROCK TRACETERATURE	1900°F
Modified NASA Modified NASA Modified NASA Modified NASA	2222 2322 2322 2322 2322 232 232 232 23	AS CAST AS CAST AS CAST	ROOM ROOM 1900 1900(X)	75.98 6.93.6	.0309 .0327 .0372 .0293	51,300	2151 1780	1230 760	67,700 54,400	33,100 25,900 (x)	1.0	1.5
NASA		rolled at 1500°F 25%	1900	93.8	.02085			1060		50,839		0.0
NASA		rolled at 1750°F 60%	1900	58.0	.01206			106		8,800		3.0
NASA NASA		as rolled	1900	55.0	.02035			700 780		34,400 37,700		0.0°
		rolled		13.8(s)	.006839	189,400	i i		226,600		ć	
NASA 143	2 4 2 4 2 4	(.0) +	F00F	22.3(T)	.01105	117,200	0661		140,300) V	
		rolled	200	24.2(S)	.008134	150,000	000		170,900		•	
NASA 148	418	(.0) +	ноом	26.0(T)	£2800°	136,500	1390		159,000		? .	
		rolled		24.5(8)	.01378			t		57,700		
NASA 148	\$ 4 (8	(.0) +	1900	29.5(T)	.01658			£		48,000		t.
		rolled	000	16.0(s)	41010.			176		7,800		c -
NASA 142	‡ *	(.0) +	2061	19.0(T)	.01204			Ç		32,400		?
CAL	गटग	rolled	190	11.0(S)	.006925			345		49,800		0.0
		(**)	}	18.2(T)	.01134			\		30,400		•

	Slongation (Percent)	ROCH 1900°F	ERATURE	2	C L	>	4	O	-	4	(O. N	i.	د. 7	(3.0	1	7.0	i	•
	ULTIMATE SI TENSILE STRENGTH	SI) 1900°F		700,121	004,791	144,100	174,600	129,300	004,74	42,800	42,000	49,500	46,500	43,300	175,200	126,400	48,700	43,600	47,000	37,200
	ULTIMATE TENSILE LOAD		•		791			505	34	405	i	ره)	222	0,0		126,		615	Ş	5
Continued)	ULTIMATE YIBLD STRENGTH		129,000	116,600	135,200	98,700	143,100	105,900							128,300	92,500				
APPENDIX G (Continued)	AREA	(sd. inches)	.007731	.008550	.006635	60600.	.003236	.00437	.00981	.01086	.01820	.01545	.01236	.01327	.00653	90600*	.01675	.01871	.01074	.01356
	THICK- NESS	(0.001")	14.1(S)	15.6(T)	13.5(s)	18.5(T)	10.0(S)	13.5(T)	14.0(S)	15.5(T)	25.8(T)	21.9(8)	21.8(S)	23.4(T)	18.6(s)	25.8(T)	24.0(S)	26.8(T)	19.0(8)	24.0(T)
ļ	TENT TEMPER- ATURE	(°F)	a C	NOON.	MOOR	FIOOR		FOOM	000	7600	•	1900	000	2064	NOO a	1001	000	200	0001	3
	CONDITION		rolled	(;) +	rolled	· +	rolled	;; +	rolled	(· -	rolled	(°,) +	rolled	(:.) +	rolled	(rolled	(B) +	rolled	(::)
	MELT NUMBER		000	200	287	રે જ	ţ	, 26	t o	200	Ç	S S	200	રે	285	ò	unc	o o	प्टग	į
			421	†	ולאנ	ţ,	į	154	ų.	174	ļ	5	166	007	נאנ	7	646	707	153	2
	ALLOY & ROLL NUMBER		o o w	ACAN	o o o	ACAN		NASA	202	ACAN		NASA	000	VCV.	4 P 4 L	vov.	o o o	ACHN	8 8 8 8	4

				TEST		APPENDIX G (Continued)	inued)					
ALLOY & ROLL NUMBER	ROLL	Mele Number	CONDITION	TEMPER- ATURE	THICK- NESS	AREA	ULTINATE YIELD STRENOTH	ULTIMATE TENSILE LOAD	ATE LE	ULTIMATE TENSILE STREMOTH	ELON (FE	ELONGATION (PERCENT)
				3		(a)	HOOM	ROCH 1	.	ROOM 1900°F	ROCH	(1900°F TURE
NASA	109	418	rolled	ROOM	50.7	.01325	153,600	2165	•	163,400	1.0	•
NASA			rolled	1900	47.0	.0179			580	32,400	_	10.3
NASA			rolled	1900	50.0	.0200			955	001,74	_	11.5
NASA			rolled	1900	46.0	.0150			535	35,700	_	٥٠ ټخ
NASA			+ (4.) + (6.)	1900	0.64	.01656			715	43,200	•	14.0
NASA	125	385	rolled	1900	41,2	.01482			540	36,400		8.0
NASA	3115	418	rol'	1900	50.5	.01758			522	29,700		15.0
'ASA	123		rolled	ROOM	0.94	.01598	149,200	2590	7	162,100	2.0	
NASA	115	418	rolled	1900	50.8	•01369			388	28,300		15.0
NASA	125	385	rolled + (E.)	1900	41.9	.01453			482	33,200		6.0
MASA	123		rolled	ROOM	42.6	.01374	138,600	2260	Ä	164,500	3.0	
TASA	125	385	rolled	1900	6.04	.01513			522	34,500		3.0
MASA	115	418	rolled	1900	η • 6η	.01857			809	32,700		0.0
NASA	123		rolled + (D.)	ROOM	42.5	.01481		1900	Ä	128,300	2.0	
:ASA	171	419	rolled	1900	54.0	.02180		-	875	40,100		12.0
NASA	166	. 5	rolled	1900	54.0	51 610.			502	26,300		1
NASA	120	462	rolled	R00f:1	54.6	.02005	120,200	2655	Ħ	134,400	1.0	
.IASA	1/1	419	rolled	1900	52.5	.02225		``	856	36,1500		15.0
MASA	122	£2 .	+ (f.) rolled + (P.)	1900	48.2	.01591		•	582	36,600		0
MABL	125	385	rolled	ROOM	41.6	.01083		1568	4	144,800	6.0	
Nása	85	385	rolled + (H.)	ROOM	39.1	.01025	123,400	1495	7	145,900	3.0	

			TEST		APPENDIX G (Continued)	(Continued)			
MELT NUMBER		CONDITION	TEMPER- ATURE (°F)	THICK- NESS (.001")	AREA (sq. in.)	日日	ULTINATE TENSILA LOAD (#)	ULTIAL TENSIL STABNOT (PSI)	ELONG.TION (FERCENT)
							ROOM 1900°F TEMPERATURE	ROOM 1900°F TEMPERATURE	ROCM 1,900°F TEMPERATURE
462		rolled	ROOM	58.3	.0184g	134,400	2990	161,700	1.0
425		rolled	чоон	51.1	£1610.	126,400	27.65	144,200	1.0
425		+ (%.) rolled + (%.)	130014	55.1	.02021	124,700	2750	135,100	1.0
ħ2ħ		rollea + (´.)	ROOM	15.8(S) 23.3(T)	.007373	207,000	1730	217,000	0
45 4		rolled + (2.)	ROOM	17.0(S) 15.1(T)	.008434 .0054E	165,00	1450	172.000	0
410		rojječ	1500	30.6	.00 20		320	34,800	0.0
413		+ (%.) rolled + (%.)	1500	5.44	.01368		555	000,04	0.4
416	•	rolled + (Q.)	1900	43.1	.01864		047	30,700	<i>G**</i> 7
425		rolled + (Q.)	1900	18.5(s) 19.5(s)	.00916		415	45,300	3.5
	7	rolled + (Q.)	1900	18.8(s) 23.2(T)	.01324 .01633		019	50,600	2.0
	H + H +	rolled + (N.) rolled + (M.)	ROOM 1900	41.2 45.1	.01467		1775	121,000	0.3
	ř +	rolled +(M.)	1900	42.0	.01461		362	24, ≥00	12.0
	Ģ.	rolled	1900	19.3	6 4 900°		27.5	41,900	3.0
	+ Ñ +	olled	1900	20.7	90200		172	24,400 (Y)	(Y) 3.0
	+ 4 +	rolled + (AA)	1900	27.3	.00926		272	29,400 (Y)	(Y) 2.0

	ULTIMATE ULTIMATE ELONGATION TENSILE TENSILE (FENCENT) LOAD STRENGTH	(#) (PSI) ROOM 1900*F ROOM 1900*F TEMPERATURE TEMPERATURE	2200 136,500	2800 143,700 2.0	2345 144,900	494 31,300 8.0	652 34,300 10.0	534 30,400 3.0	2130 131,500 1.0	1560 136,500 0	434 36,500 1.0	2035 135,800 0	3400 136,700 2.0	3280 119,300 2.0	1740 63,600 40,700 2.0 1.0	3160 970 117,900 37,000 2.0 10. 0	2145 305 103,600 41,900 2.0 5.0	2750 2.0 109,000 28,400 2.0 4.5	1900 1155 71,600 53,200 - 1.5	2390 91,600 - 10.0 725 27,100 - 10.0	680 24,300 2.0	
	CTIMATE ENSILE FRENOTH		,500	3,700	006*+	31,300	34,300	30,400	1,500	96,500	36,500	5,800	002,91	9,300							24,300	
		1,006				ħ6 1	652	534			1 87				1040	970	905	756	1155	735	680	
ıtimed)	ULTIMATE U YIELD T		121,900 22	120,600 28	123,300 2				123,500 2.	21		ĸ	125,400 34	110,700 3	11	112,700 33	103,600 21	12	19	. 23		
APPENDIX G (Continued)	AREA	(sq. in.)	.01612	.01999	.01618	.01579	.01899	.01756	.01621	.01143	•110	.01499	.02468	.02778	.02557	.02680 .02622	.02071	.02524 .02663	.02654	.02609	₩51 Z 0°	1 1
		(,001,)	41.2	49.5	41.0	40.3	48.3	45.1	7.5	9.44	37.3	2.74	97.5	110.6	108.4	102.3	82.1 85.4	103.0	106.3 80.8	104.1	110.0	
ı	temper- Temper- Ature	(•F)	ROOM	ROOM	ROOM	1900	1900	1900	ROOM	ROOM	1900	ROOM	Room	Room	1900 Room 1900	Room 1900	Room 1900	ROOM 1900	ROOM 1900	1900 1900	1900	
	CONDITION		rolled	+(N.) rolled	+(N.) rolled	+ (N.) rolled	rolled	+ (N.)	+ (N.) rolled + (0.)	rolled	+ (I.)	+ (I.) rolled + (F.)	as cast		as cast as cast as cast	as cast as cast	(a.)	as cast as cast	as cast as cast	as cast as cast	as cast	1
	MELL NUMBER		415	± %				425	387		385	418	1 01	80	200	ผผ	01 01	20.00	୭.୭.	ળ ળ	ღ	
	ALLOY & ROLL NUMBER		NASA 114	NASA 122				NASA 122	NASA 119	NASA 123	NASA 125	NASA 109	INCO 713C		M. Modified 390 M. Modified 390 INCO 713C: 390	(392 2(Ta-C)	Mcdified 392 INCO 713C 392	Ta-C-Mo modified428 INCO 713C 428	Ta-C-Mo-W 429 modified INCO 429 713C	Zr-C modified 432 INCO 713C 432	T1-C modified 433 INCO 713C	

***************************************			TEST		APPENDIX G (Continued)	(Continued)		ļ				
ALLOY & ROLL NUMBER	MELT NUMBER	CONDITION	TEMPER- ATURE	THICK- NESS	AREA	ULTIMATE YIELD Smoonsmu	ULTIMATE TENSILE	ATE LE	ULTIMATE TENSILE	ម្តីស	ELONG (PER	ELONGATION (PERCENT)
			(•F)	(,001")	(sq. in.)		LOAD (#) ROOM TOPERATURE	1900 °F	STRENGT (PSI) ROOM TEMPERATURE	1900 .r	ROOM TEMPERATURE	1900°F
Cr-T1-C modified436 INCO 713C 436	36 436	as cast as cast	ROOM 1900	118.3	.02929		2570	1155	90,500	39,400	1.0	1.5
Cb-Ta-W-C modified INCO 713C	437 437	as cast as cast	ROOM 1900	112.2 93.8	.02797 .02457		3255	845	116,400	34,400	1.0	2.5
Al-Ti modified INCO 713C	438	as cast	ROOM	113.1	.02737		2940		107,400		1.0	
Ta-C-Mo-W modified INCO	457 457	as cast as cast	ROOM	100.4	.02155			1260 1350		58,500		0,0
fled	388	rolled at R.T. 20%	1900	86.7	.01932			200		25,900		0.4
TNCO /13c	8	rolled at 1800°F 60%	1900	40.3	.00951			272		28,500		4.0
INCO 713C INCO 713C		as rolled as rolled	1900	44.0 41.0	.0150			625 615		41,778 43,991		0.0 mm
INCO 713C	1 0 1	rolled at	1900	82.8	.01942			930		47,900		c c
INCO 713C	1 05	rolled at	1900	82.5	.01948			810	~	41,600		3.0
Ta-C-No-W Modified INCO 713C	157	rolled 60% at 2100°F	1900	8.74	.01024			130		12,700		5.0
	550	rolled + (J.)	1900	9*91	.02299			1165		50,700		5.0
INCO 713C 216	550 550	rolled + (J.) rolled	1900	46.0 45.1	.02585			1290 1265		49,900		8.0
		(:)						ı	•	48,900		5.0
7130	548	rolled + (J.)	1900	51.7	.03108		•	1370	7	001 44		c c
	248	rolled + (J.)	1900	52.0	.03161		-	1325	* #	, 1000 F) (
INCO 713C 210	248	rolled	1900	50.1	.02967			076	•	200		0.0
		7.5.					•	<u> </u>	#	42,800		10.0

	ELONGATION (PERCER)	HOOM 1900°P	12.0	10.0	6.0	8.0	g. 0.		6.0	13.0	12.0	10.0	5.0	15.0	7.0	8.0	6.0	0.0	0.4	12.0			12.0	10.0	0.9	•	6.0
		1900•1	46,500	50,800	50,000	45,300	006,74	3.0	31,500(Y)	24,700(Y)	30,700(Y,	45,700	57,000 (z)	69,700(2)	0,900	65,300(2)	004,54	47,800	45,300	004,24			500	42,800	28,800	300 14.0	33, 200
	SHE	1900°F	1470	1345	1350	1205	1235	001,951	021	356	ŧ,	7695	2100	2580	0# 		1728	1800	805	940	ま1	08 1	178,200		28	164,200	
	ULTIMATE TENSILE LOAD	(#) Room Temperature						0141							hefone	010100							3130	2010			
(þe	ULTIMATE YIELD STRENGTH	(PSI) ROOM TEMPERATURE						141,200							אייהשטי הייים אפיים הפאן ביה לייור *	Tar was move								124,600			
APPENDIX G (Continued)	A REA	(sq. inches)	.02969	67920.	.02699	.02660	.02576	.00903	.01333	.01439	.01643	.03703	.03684	66980	.01065	10108	.03725	.03769	.0178	.0170	.01762	.01667	.01756	.01224	.01544		
	THICK- NESS	(.001")	9.05	46.5	4.74	47.2	45.9	18.1	17.8	18.5	20.8	55.7	55.6	55.2	19.2	19.3	55.8	8.8	0.44	45.0	51.8	50.8	46.2	50.6	46.1		
	TEMPER- ATURE	(.F.)	1900	1900	1900	1900	1900	ROOM	1900	1900	1900	1900	1900	1900	1900	1900	1900	1∫00	1900	1900	1900	1900	ROOM	ROOM	1900		
	CONDITION		rolled	+ (K.) rolled	+ (K.)	rolled	+ (L.) rolled + (L.)	rolled	+ (U.)	rolled	rolled	rolled	rolled	rolled	rolled	rolled	rolled	+ (36) + (33)	rolled	rolled	rolled	rolled	rolled	rojled	rolled	+(¤•)	
	Melt Number		548	548	550	550	550	550	055	550	550	245 1	1 547	1 547	1 550	1 550	1 547	245	•		‡0 ‡	1 01	707	1 0†	†O†		
	ALLOY & ROLL NUMBER		INCO 713C 210	INCO 713C 210	INCO 713C 216	INCO 713C 216	INCO 713C 216	INCO 713C 231	INCO 713C 231	I.100 7130 230	INCO 713C 230	INCO 713C 211	INCO 713C 211	INCO 713C 211	INCO 713C 231	INCO 713C 231	INCO 713C 211	INCO 713C 211	INCO 713C -	INCO 713C	INCO 713C 122	INCO 713C 122	INCO 713C 122	INCO 713C 122	INCO 713C 129		

					APPENDIX G (Continued)	:tmed)			
ALLOY & ROLL MUMBER	MELT NUMBER	CONDITION	TEMPER- ATURE	THICK- NESS	AREA	ULTIMATE YIELD	ULTIMATE TENSILE	ULTEARTE TENSILE	ELONGATION (PERCENT)
			(& E)	("100")	(sq. inches)	STRENGTH (PST) ROOM TEMPERATURE	LOAD (;;) ROOM 1900*F FEFFRATURE	190	ROOK 1900°F
INCO 713C 129	1 04	as rolled	ROOM	42.5	.01455	135,700	2725	187.300	18.0
INCO 713C 129	1 04	as rolled	1900	45.5	.01638		842		10.0
INCO 713C		(8)	ноом	31.2	.009348	105,700	1352	1,4,600	14.9
INCO 713C 182		rolled + (C.)	ROOM	30.1	58600.	143,300	1840	196,800	12.0
INCO 713C 193		rolled	ROOM	9.74	.02054	123,700	3500	170,400	14.0
INCO 713C 193		rolled + (C.)	ROOM	44.3	.01830	121,900	3220	176,000	15.0
INCO 713C 191		rolled	ROOM	41.2	.01387	122,500	2360	170,200	20.0
INCO 713C 191		rolled	ROOM	40.0	.01348	123,500	2300	170,500	19.0
INCO 713C 191		rolled + (H.)	КООМ	42.0	.01386	123,700	2455	177,100	15.3
(A.) HT 16 HRS at HT 24 HRS at D. HT 24 HRS at HT 3 HRS at HT 1/2 HR at HT 1/2 HR at HT 64 HRS at N.) HT 24 HRS at 1.	at 2000 at 2200 at 2200 at 2200 at 2200 if 2150 at 2200 if 2150 at 2200 t 1750,	IT 16 HRS at 2000, water quenched. IT 16 HRS at 2200, water quenched. IT 24 HRS at 2200°F air cool. IT 3 HRS at 2200°F air cool. IT 1/2 HR at 2200°F air cool. IT 4 HR at 2200°F air cool. IT 4 HR at 250°F air cool. IT 4 HR at 250°F air cool. IT 4 HR at 250°F air cool. IT 4 HR at 2150°F air cool. IT 4 HR at 2200°F air cool. IT 4 HR at 2200°F air cool. IT 64 HRS at 2200°F air cool. + 61 HR at 154 HRS at 150°F air cool. IT 64 HRS at 2200°F air cool. + 64 HRS at 1750, air cool.	quenched. 'quenched. cool. cool. ibed in 170°F o cool. cool. 24 HRS a cool. dl. dl. cool. dl. dl. dl. dl. dl. dl. dl. dl. dl. d	HRS at 1600°R air cool. HR at 1600, air cool. HR at 1600, air cool. HRS at 1450°P air cool. HRS at 1450°P air cool. HRS at 1600°P air cool.	(F.) (G.) (G.) (G.) (G.) (G.) (G.)	HHT COLUMN MAIN TO THE THE HHT THE HHT TE THE THE THE TE THE TE THE TH	HT 24 HRS at 2200°F, air cool + 54 HRS at 24 HRS at 1750, air cool + 2 1/2 HRS at 2200, retort cool. 64 HRS at 2200, retort cool. 64 HRS at 2150°F air cool + 64 HRS at 144 Thickness measured with flat micrometer. HT 61 HRS at 2150°F air cool + 24 HR at 17 61 HR at 2150°F, air cool + 24 HR at 184 at 180°F, air cool + 24 HR at 184 at 184 at 184 at 186 at 187 mum load rate, head travel about .01° HR 64 HR at 220°F, head travel about 5.0 HT 64 HR at 220°F air cool + 54 HR at 1800°F, HT 64 HR at 220°F, the 64 HRS at 1575°F. HT 64 HR at 220°F air cool + 24 HRS at 1600°F, 1900°F.	64 H3S at 1 RS at 1450 morrometer. THA at 160 H HA at 175 H HA at 175 HA at 1050 bout 5.0 /m to 160°F, at	1500°F air cool, 2000°F air cool, air cool. 60, air cool. 6°F, air cool. minute, length of minute, length of recol + 1/2 IR at

Ħ. III. 4 APPLICATION. Final report, Apr 63, 1499 incl Agromentical Systems Division, Dir/Materials & Processes, Metals & Cermics Lab, Wright-Patterson AFB, Chio Bpt No. ASD-IIB-62-869. DEVILOPMENT OF A MICKEL BASE ALLOT SHEET FOR HIGH IMPERATORS illus., tables.

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Contract AF 33(616) research AFSC Project 7351, Task 735105

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Hokel base alloys Hgh temperature In ASTIA Collection

f. J. Riley Aval fr OTS

The objective of this contract was to develop 15 to 30 mil nickel alloy sheet having 50,000 psi tensile strength at 1900°F, having good correcton (exidation) resistance, and good ductility. This objective was essentially attained by developing a new process of directly rolling thin east slabs of mickel base alloy into about on a specially designed right rolling mill. The pre-existing mickel base east-ing alloys and a series of experimental com-

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ing alloys were initially investigated in the as east condition in this program. The two starting alloys were lase 713c and MAA's That alloy. Of the new experimental compositions, alloy in 429 (a Ta-W-C-Or modified lase 713c) has 1900°F tenails strength exceeding 50,000 pai in the as east condition, as does the Taff alloy. Inco 713c has a tensile strength of about 40,000 pai in the as east limited time at 1900°F; that of No. 429 alloy is substantially better; and that of 1000 7130 is best of the three. tion resistance of Talk alley is adequate for positions obtained by modifying the two start good room temperature ductility. The oxidacondition. Bot rolled Inco 713e indicated

APPLICATION. Final report, Apr 63, 149p incl & Processes, Metals & Ceramics Lab, Wright-Patterson AFB, Ohio Rpt No. ASD-TDR-62-869. DEVELOPMENT OF A MICKEL BASE ALLOY SHEET FOR HIGH TEMPERATURE Aeronautical Systems Division, Dir/Materials illus., tables.

Unclassified report

The objective of this contract was to develop 15 to 30 mil nickel alloy sheet having 50,000 psi tensile strength at 1900°F, having good corrosin (oxidation) resistance, and good cutility. This objective was essentially attained by developing a new process of directly rolling thin cast slabe of mickel base alloy into sheet on a specially designed rigid rolling mill. No pre-existing nickel base cast-ing alloys and a series of experimental com-

tion resistance of Ta28 alloy is adequate for limited times at 1900°F; that of No. 4.29 alloy is substantially better; and that of Inco 7130 tions, alloy No. 429 (a In-M-C-Cr modified Inco 71.2c) has 1900°F tensile strength exceed-ing 50,000 pai in the as east condition, as does the TaZ8 alloy. Inco 713c has a tensile strength of about 40,000 pai in the as east positions obtained by modifying the two starting alloys were initially investigated in the as cast condition in this program. The two starting alloys were inco 713e and MASA's Ta28 alloy. Of the new experimental composigood room temperature ductility. The oxidacondition. Not rolled Inco 713c indicated is best of the three. Φ

Mickel base alloys High temperature 44

Contract AF 33(616) MFSC Project 7351, Mask 735105 H. H

Dallas 22, Texas H. Greenswald, Jr. T. J. Hiley Aval fr OTS Chance Vought Corp -188 III. ï.

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